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The detection of crevice corrosion in titanium and its alloys through the use of potential monitoring

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through the use of potential monitoring**

Bergman, Dave Dror, M.S.

University of Nevada, Las Vegas, 1992

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THE DETECTION OF CREVICE CORROSION IN TITANIUM AND ITS
ALLOYS THROUGH THE USE OF POTENTIAL MONITORING

by

Dave D. Bergman

A thesis submitted in partial fulfillment
of the requirements for the degree of

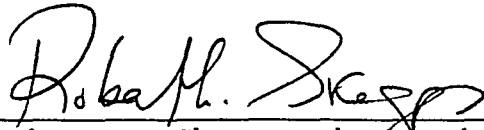
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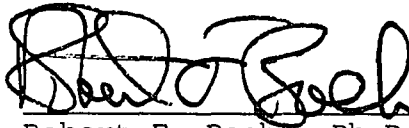
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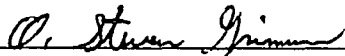
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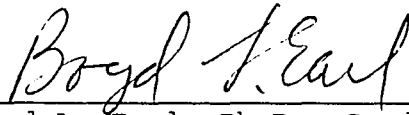
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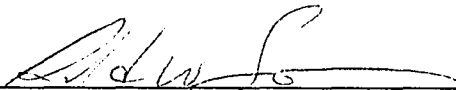
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ABSTRACT

Conventional laboratory crevice corrosion testing requires approximately thirty days and is a simple go/no go test which does not indicate crevice incubation time and propagation. This study focuses on the effectiveness of potential monitoring to detect the onset of crevice corrosion and propagation for several titanium alloys in chloride environments. Utilizing this technique, crevice corrosion initiation is determined after several days, thus a shorter test period is required. This technique may well be suited for field as well as laboratory applications. Crevice corrosion incubation periods and corresponding potential behavior are investigated along with the effect of crevice to uncreviced surface area ratio. Under freely corroding conditions, which better simulate in-service conditions, the relationship between the corrosion potential (E_{corr}) and crevice corrosion initiation and propagation was readily determined. Corresponding crevice corrosion initiation and steady state propagation surface morphology was also examined.

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INTRODUCTION

Corrosion is defined as the deterioration of a material (usually a metal) or its properties because of reaction with its environment. Corrosion can be classified into two categories: 1) aqueous corrosion, and 2) dry corrosion. Aqueous corrosion, discussed in this study, occurs when a liquid is present, which accounts for the greatest amount of corrosion. Dry corrosion occurs in the absence of a liquid phase, and is associated with high temperatures, such as corrosion in vapors and gases. Thermodynamic calculations provide information on whether or not the corrosion reaction is possible. That is, corrosion will not occur unless the spontaneous direction of the reaction indicates metal oxidation. However, even if the corrosion reaction is possible it does not mean that corrosion will occur, since the reaction may proceed at a negligible rate. Thus, the major interest is in the kinetics or rate of corrosion.

Virtually all aqueous corrosion processes can be classified as the oxidation of a metal to its ion - being the partial anodic reaction. The partial cathodic reaction which involves a decrease in valence charge or consumption of electrons occurs simultaneously and at the same rate on the metal surface. The most common cathodic reactions involve the reduction of oxygen and hydrogen, since acidic media in contact with air is often encountered.

General aqueous corrosion is the most common form of corrosion, and is characterized by a uniform attack over the entire metal surface. Crevice corrosion is localized corrosion resulting from the formation of a concentration cell in a crevice formed between a metal and a nonmetal, or between two metal surfaces. Crevice corrosion in an aqueous chloride environment occurs due to oxygen-depleted reducing acid conditions developing in tight crevices. Dissolved oxygen or other oxidizing species are consumed faster in the crevice than diffusion from the bulk solution can replenish them. As a result, metal potential in the crevice becomes active (negative) relative to the metal exposed to the bulk solution. This creates an electrochemical concentration cell in which the crevice becomes anodic relative to the cathodic surface outside the crevice. This tends to produce an excess of positive charge in the crevice solution, which is balanced by the migration of chloride ions into the crevice. Hydrochloric acid is then produced inside the crevice, lowering the pH level gradually. When a critical pH level is reached crevice corrosion will initiate.

The titanium industry, has traditionally relied on a thirty day go/no go freely corroding crevice test assembly in chloride environments at 90° C and above.¹ The primary goal was to provide information on maximum safe operating temperature at a given chloride level and pH. In addition, there have been several studies of potential-time behavior of freely corroding crevice and plain specimens of unalloyed titanium. However to date alloyed titanium behavior has not been studied. M. Kobayashi² observed a steady state potential of -465 mV SHE (Standard Hydrogen Electrode) after 40 days in a boiling, 6 Wt% NaCl bulk solution with a crevice specimen containing an NaCl crystal sealant and a crevice/uncrevice ratio of 1:1. The freely corroding initiation/propagation potential criteria, however, was not explored. P. McKay³ reported approximately -300 mV SHE as a steady state potential at 150° C, 1.6 Wt% NaCl solution, and a crevice/uncrevice ratio of 1:4. The freely corroding initiation/propagation potential criteria was not quantified, but incubation periods to a steady-state potential were reported as generally 10 to 15 hours at 150° C, and 20 to 30 hours at 95° C.

A study of current-time behavior of freely corroding crevice and plain specimen was initially reported by T.S. Lee⁴ for stainless steel. A crevice corrosion cell based on the concept of a galvanic couple consisting of a crevice anode and a boldly exposed comparably large cathode (typically anode/cathode ratio 1:90) with provisions for measurement of a current between the two was used. Lee observed that an increase in current did not correspond to crevice initiation as determined visually. However, the mean galvanic current measured throughout the test duration was in excellent agreement with corrosion rate using weight-loss data and provided good propagation data. Utilization of this technique in the present study, has shown that a current increase was consistent with crevice surface oxidation, on ASTM grade 2 titanium, after a two day immersion in 5% NaCl, PH 2, 93° C solution. However, several studies of similar galvanic coupling of grade 2 in chloride solutions, including the one by P. McKay³, reported that after the galvanic current reached a maximum (25-75 hours) it continuously decreased with time, eventually reaching a low constant value, although crevice propagation continued. This current decrease could be attributed to an increasing potential balance between such galvanically coupled specimens. In addition, current-time monitoring techniques of freely corroding crevice specimen are generally not as practical as potential monitoring, since they require a more complex set-up, such as constant monitoring with a zero-impedance ammeter.

It is important to establish a practical method to detect the onset of crevice corrosion and propagation so that meaningful predictions of crevice corrosion behavior of alloyed and unalloyed titanium may be made. This investigation has shown that laboratory potential monitoring is a relatively simple method for quantifying the initiation and propagation stages of crevice-corrosion.

This potential monitoring technique could prove useful as an on-line monitor of crevice corrosion in industrial processes, monitoring both a prepared crevice cell and/or the actual subject structure. Before this can be achieved, however, it is necessary to further quantify the potential behavior at initiation and propagation stages of crevice corrosion, with regards to the effect of crevice/uncreviced ratio, and oxidizers.

EXPERIMENTAL

Materials

The compositions of all the alloys used in this study are listed in Table 1. Titanium specimens were from mill and laboratory produced sheet, (Ti-Pd, Beta C buttons) with standard annealing treatments ranging from 1450°F for the alpha and near alpha alloys, to 1550°F anneal for the beta alloys. Nickel based alloys were tested in the standard annealed condition. Specimen sheet gage varied from 0.03 to 0.06 inch. Titanium specimens were tested in the as-pickled surface condition, and nickel based alloys were abraded to a 240 grit finish. Preparation of pickled titanium specimens involved immersion of 5 minutes in a bath containing 35 vol. % stock nitric acid, 5 vol. % stock hydrofluoric acid, and distilled water. After pickling, the specimens were rinsed in distilled water and air dried. All test solutions were prepared with distilled/deionized water and reagent grade chemicals.

Apparatus

The electrochemical cell used is shown in Figure 1. The Huey corrosion test apparatus used for near boiling (93-98°C) testing is shown in Figure 2. A high temperature Ag/AgCl reference electrode was used for all potential measurements. The reference potential of the Ag/AgCl/KCl (0.1 N) electrode is 0.244 V SHE at 93°C. Corrosion potential measurements were made using a Triplet Corporation 3360 electrometer. The electrode/electrometer system has a reported accuracy of ± 1.0 mV in the 0 to ± 2000 mV range.

Procedure

The standard test condition consisted of testing a crevice assembly with a 4:1 crevice/uncreviced surface area ratio in a 5 wt.% NaCl solution at pH 2, 93°C, and natural aeration.

A crevice was formed by compressing the test specimen between two Teflon sheets torqued to 1.8 N-m, utilizing two plates of grade 7 titanium and grade 2 titanium bolts wrapped with Teflon tape to ensure electrical insulation. The crevice specimens were assembled under the bulk chloride solution to avoid conditions of inconsistent wetting of the creviced surfaces. The standard creviced area was 1 inch square and the external uncreviced area was 1/4 inch square, thus, achieving a 4:1 crevice/uncreviced surface area ratio. The 1:3 and 1:90 crevice/uncreviced surface area ratios were composed of a creviced area of 0.3 inch square and the external uncreviced area was 1 inch square and 27 inch square respectively. A Teflon insulated grade 2 titanium wire was resistance welded to the uncreviced area for monitoring purposes. The nickel based alloys were assembled similarly, except that plates of alloy 625 and Teflon insulated nickel wire were utilized. Post test evaluations of titanium crevice specimens required a light (5-8 sec) sandblasting per ASTM G 46, to remove the tenacious corrosion product scale and facilitate examination of the crevice surface. Nickel based alloys corrosion product scale was removed using a paper fly-wheel with 240 grit abrasive. Crevice specimen surfaces were examined at 8X magnification.

RESULTS

Potential Monitoring Reproducibility

To determine potential indication reproducibility 10 creviced titanium grade 1 specimens and 10 titanium grade 2 specimens were tested at standard* conditions. Crevice specimen potentials were recorded at 3 to 24 hour intervals and certain corresponding creviced surfaces examined in 10 to 100 mV variations. In addition, specimens at matching potentials were compared. Crevice initiation was defined to begin when crevice pits were first detectable at 8X magnification. Crevice propagation was defined to begin when a steady-state potential was first reached after initiation had occurred. When a potential lower than -360 mV (Ag/AgCl) was recorded crevice corrosion initiation had occurred on all specimens observed. When a potential higher than -290 mV was recorded crevice initiation has not occurred on any of the specimens observed. However, at magnifications of 16X and higher, crevice pits were detected on two specimens at higher potentials: ranging from -150 to -290 mV. At potentials ranging from -290 to -360 mV crevice initiation was observed on some of the specimens, whereas others only exhibited a thicker oxide film, typical by its variety of colors (blue, violet, etc.). Specimens examined at potentials varying by 5-10 mV exhibited comparable surface morphology. After crevice initiated a steady-state potential of -440 mV with approximately 40 mV daily fluctuation was reached on all specimens, and corresponding severe crevice corrosion was noted. Table 2 (a) lists the potentials recorded, and Figure 10a,b,c, and d illustrates the corresponding creviced surface observed.

Note: The "free surface" referred to in the tables and graphs means the uncreviced specimen.

Unalloyed Titanium Potential/Time Behavior

Test Condition: Standard

The corrosion potential/time behavior observed on grade 1 and grade 2 titanium creviced specimens differed extensively from plain specimens. The plain specimens exhibited typical passivation behavior of a freshly exposed metal surface. Their potential (E_{corr}) initially was active, typically -150 mV, and then increased because of increasing surface oxidation finally reaching a constant value of 400 to 440 mV after 3-4 days. On creviced specimens, however, the potential remained active, initially -200 mV, and then decreased to -320 mV when crevice corrosion initiation was first observed on grade 1 and -340 mV when initiation was first observed on grade 2. Minimum incubation periods for initiation were approximately one day less for grade 1 than for grade 2, and were 3 and 4 days respectively. Steady-state propagation potentials were similar for both grade 1 and 2 (-440 to -480 mV) and were reached generally after 10 days. The small 40 mV fluctuations in propagation potentials are believed to be due to new crevice formations

*Standard test conditions are defined under Experimental Procedure.

occurring as propagation progresses. Grade 1 specimens exhibited more severe crevice corrosion than grade 2 after a 30 day testing period, which was in agreement with their initiation behavior. The averaged potential-time behavior, of five specimens of each grade, given in Table 2 (b), is shown in Figure 3a and 3b. Figure 11a, and b illustrates the corresponding surface observed.

Test Condition: Standard With Air Sparging

The plain specimens exhibited accelerated passivation in this high dissolved oxygen concentration solution. The potential (E_{corr}) initially decreased to active values but then increased rapidly, reaching a constant value of 440 to 470 mV after less than one day. The potential remained active on the creviced specimens continuously decreasing to initiation and propagation potential values similar to those in the naturally aerated solution. However, minimum incubation periods for initiation were accelerated by approximately one day for both grades. Propagation potentials were similar for both grade 1 and 2 and steady-state was reached generally after six days. A more severe crevice corrosion attack was noted on both grades after 30 days in comparison to that observed in the naturally aerated solution, with grade 1 exhibiting the more severe attack. The averaged potential-time behavior, of four specimens of each grade, given in Table 3, is shown in Figure 4. Figure 12a, and b illustrates the corresponding surface observed.

Test Condition: Standard With 1000 ppm Fe+3 (Ferric Chloride)
in Bulk Solution, Crevice Specimens Assembled Under a pH
2 Solution Without Fe+3.

The plain specimens exhibited accelerated passivation in this highly oxidizing solution. The potential (E_{corr}) initially decreased to active values but then increased rapidly, reaching a constant value of 580 to 630 mV after less than one day. The potential was active on creviced specimens initially, but then increased to 550 mV after one day. A potential of 515 mV was recorded when initiation was first noted on grade 1, and 505 mV on grade 2. Propagation potentials were 300 mV \pm 50 mV for both grades. Minimum incubation periods to initiation and steady-state propagation were accelerated similarly to those observed in the air-sparged solution. Crevice corrosion severity was also comparable to that observed in the air sparged solution. When crevice specimens were assembled under the bulk solution (containing Fe+3) crevice corrosion did not occur. The averaged potential-time behavior, of three specimens of each grade, given in Table 4, is shown in Figure 5. Figure 13a, and b illustrates the corresponding surface observed.

Test Condition: Standard at pH 5

The plain specimen behavior and creviced specimen initiation and propagation potentials were similar to the pH 2 naturally aerated solution. However, minimum incubation periods to initiation and propagation were longer; 6 days for grade 1 and 7 days for grade 2 to initiation, and generally 17 days to reach steady-state propagation. Accordingly, less severe crevice corrosion

was noted after 30 days, with grade 1 exhibiting more severe crevice corrosion than grade 2. The averaged potential-time behavior, of three specimens of each grade, given in Table 5, is shown in Figure 6. Figure 14a, and b illustrates the corresponding surface observed.

Test Condition: Standard With a Crevice/Uncreviced Ratio of 1:3

This larger cathode (uncreviced area) to anode (creviced area) ratio had a pronounced effect on accelerating minimum incubation periods to crevice initiation and steady-state propagation. Minimum incubation periods for initiation were one day for grade 1 and steady-state propagation was reached, generally, after 4 days. Initiation and propagation potentials were similar to those observed for the 4:1 ratios. After 30 days immersion, more extensive crevice corrosion (with some perforation) than that observed in the presence of oxidizing species was noted. The potential-time behavior of three specimens is given in Table 6. Figure 15 illustrates the corresponding surface observed.

Test Condition: Standard With a Crevice/Uncreviced Ratio of 1:90

This ratio had some slight effect on the potential measured with initiation being observed at -300 mV, and propagation at -440 to -500 mV for grade 1. Moreover, a more noble potential of -100 to -150 mV was recorded on day 2 after initiation occurred, and then continuously decreased until propagation potentials were reached. Incubation periods, and crevice corrosion severity were similar to those described above for the 1:3 ratio. The potential-time behavior of three specimens is given in Table 7. Figure 16a and b illustrates the corresponding surface observed.

Alloyed Titanium Potential/Time Behavior

Alloy: Titanium Grade 12

Test Condition: Standard at 95°C

pH 1

The corrosion potential/time behavior observed on creviced specimens, again, differed extensively from plain specimens. The plain specimens exhibited passivation behavior similar to that described above for unalloyed titanium. However, creviced specimens were initially active, but then partially repassivated to 100 mV after 4-5 days. Crevice initiation was observed when the potential became active again at -150 mV, after minimum incubation periods of 8 days. After crevice initiated, repassivation to 30 mV was again noted the following day. Repassivation cycles continued from -150 to 30 mV in approximately one day intervals, eventually decreasing to a range of -150 to -60 mV as crevice corrosion propagated and the full duration of the test. An active or passive potential remained constant for approximately 12-24 hours throughout the test.

pH 0.5

Initiation and propagation potentials and their behavior were similar to those observed in the pH 1 solution, however incubation periods for initiation were reduced to 5 days.

The averaged potential time behavior, of three specimens at each pH level, given in Tables 8 (a) and (b), is shown in Figures 7a and b. Figure 17 illustrates the corresponding surface observed.

Alloy: Titanium Grade 7

Test Condition: Standard at 95°C

pH 1

The plain specimens behavior was similar to the creviced specimens behavior, both exhibiting typical passivation behavior of a freshly exposed metal surface. Their potential initially was active, and then increased because of increasing passivation of the surface. However, it took 7-8 days for creviced specimens to reach a constant potential of 400 mV, whereas plain specimens reached a constant potential of 470 mV after only 3-4 days. Crevice corrosion did not occur after a 30 day testing period.

pH 0.5

A similar behavior to the one described above was noted, however it took 17 days for creviced specimens to reach a constant potential of 330 mV, whereas plain specimens reached a constant potential of 420 mV after only 4-5 days. Crevice corrosion did not occur after a 30 day testing period.

The averaged potential-time behavior, of three specimens at each pH level, given in Tables 8 (a) and (b) is shown in Figures 7a and b. Figure 18 illustrates the corresponding surface observed.

Alloy: Grade 2 with 0.01, 0.02, and 0.05 wt.% Pd.

Test Condition: Standard at 95°C

pH 1

The potential behavior of these low Pd alloys creviced specimen differed from that of the higher Pd alloy (0.12 wt.%) grade 7. Creviced specimens with 0.01-0.05 wt.% Pd exhibited a less noble potential behavior than grade 7. Their potential continuously decreased reaching -250 mV after two days, and then rapidly increased because of increasing passivation of the surface eventually reaching a constant potential of 200 mV after 10-12 days. Creviced specimens with higher wt.% Pd exhibited a slightly more noble potential behavior than those with the lower wt.% Pd. Plain specimens potential

behavior was similar to that of grade 7. Crevice corrosion did not occur after a 30 day testing period.

pH 0.5

At this pH level the potential remained active on creviced specimens eventually reaching a constant potential of approximately -250 mV after 15 days. The higher wt.% Pd specimens, again, exhibited a more noble potential than those with lower wt.% Pd. Plain specimens behaved in a similar fashion to the one described above. Crevice corrosion did not occur after a 30 day testing period, however the 0.01 wt.% Pd specimens exhibited severe general corrosion outside and inside the crevice area.

The averaged potential-time behavior of three specimens at each pH level given in Tables 9 (a) and (b) is shown in Figures 8a and b. Figure 19a, and b illustrates the corresponding surface observed.

Alloy: Ti-3Al-8V-6Cr-4Mo-4Zr, Annealed Condition
Test Condition: Standard at 95°C

pH 1

The potential behavior of plain and creviced specimens was similar to the one described for grade 12, with creviced specimens exhibiting repassivation to 60 mV after crevice corrosion initiated at -304 mV. Then, repassivation cycles range continuously decreased, eventually reaching a range of -304 to -80 mV as crevice corrosion propagated. Minimum incubation periods for initiation were 15 days. Severe crevice corrosion was noted after a 30 day testing period.

pH 0.5

Similar potential behavior to the one observed at pH 1 was noted, however minimum incubation periods for initiation were reduced to 5 days. Severe crevice corrosion was noted after a 30 day testing period.

The averaged potential-time behavior, of three specimens at each pH level, given in Tables 10 (a) and (b) is shown in Figures 9a and b. Figure 20 illustrates the corresponding surface observed.

Alloy: Beta-21S Ti-15Mo-2.7Nb-3Al-0.2Si, Annealed Condition
Test Condition: Standard at 95°C

pH 1

Crevice specimens behavior was similar to plain specimens, both exhibiting passivation behavior of a freshly exposed metal surface. However, creviced specimens passivated slowly, eventually reaching a cyclic potential

of 0 ± 50 mV after 7 days, whereas plain specimens passivated rapidly, reaching a constant potential of 450 mV after only 3-4 days. Crevice corrosion did not occur after a 30 days testing period.

pH 0.5

Similar behavior to the one described for pH 1 was noted, however, creviced specimens reached the cyclic potential of 0 ± 50 mV after 13 days. Crevice corrosion did not occur after a 30 day testing period.

pH 0.2, 98°C

At this pH level creviced specimens potential remained active, passivating to -50 mV after 5 days and then continuously dropping to -300 mV after 10 days, and repassivating again to -50 mV after 30 days. However, crevice corrosion did not occur after a 30 day testing period.

The averaged potential-time behavior of three specimens at each pH level, given in Tables 10 (a), (b) and (c), is shown in Figures 9a,b, and c. Figure 21a, and b illustrates the corresponding surface observed.

Nickel Alloys Potential/Time Behavior

Alloy: Nickel Alloy 625, Annealed Condition

Test Condition: Standard at 95°C

pH 2

The potential of plain and creviced specimens was similar, and remained active -250 to -300 mV throughout the 30 day test. Thus, crevice initiation and propagation was not detectable. Moderate crevice corrosion was observed at the end of a 30 day testing period.

pH 1

Similar behavior to the one at pH 2 was observed. The potential remained active, -300 to -350 mV, throughout the 30 day test, and severe crevice corrosion was noted.

pH 0.5

Similar behavior was again observed. The potential remained active -400 to -450 mV throughout the 30 day test, and severe crevice corrosion was noted.

Alloy: Nickel Alloy C-276, Annealed Condition

Test Condition: Standard at 95°C

pH 1

The potential of plain and creviced specimens, again, was similar, thus crevice initiation and propagation was not detectable. The potential of the specimens remained at -270 to -310 mV throughout the 30 day test, and severe crevice corrosion was observed.

pH 0.5

The potential behavior observed was similar to the one described for pH 1, with potential varying from -300 to -340 mV. At the end of a 30 day testing period severe crevice corrosion was observed.

Tables 11 (a), (b) and (c) illustrate the averaged potential of three specimens at each pH level. Figure 22a and b illustrates the corresponding surface observed.

DISCUSSION

The results illustrated that measuring corrosion potential/time profiles provided a relatively simple means of quantifying the onset and propagation of crevice corrosion on titanium. Tests concluded upon crevice initiation, provided satisfactory crevice corrosion resistance data by allowing comparison of incubation periods to initiation, thus a shorter test period was required. The reproducibility of potential measured and the extent of crevice corrosion observed suggests that this technique provides a real-time signal for monitoring crevice corrosion.

Unalloyed Titanium

Corrosion Behavior:

Critical potentials recorded for crevice initiation and propagation remained generally constant with solution parameter variations and crevice/uncreviced surface area ratio. The only exception was the addition of a high potency oxidizer - ferric chloride, which increased the corrosion potential (E_{corr}) significantly. In turn, crevice initiation and propagation potentials, which consist of a mixed crevice and uncreviced (E_{corr}) potential, increased considerably. In addition, the higher 1:90, crevice/uncreviced surface ratio had a minor effect on increasing crevice initiation potential (and near initiation potential) due to the larger uncreviced area initially raising the overall potential (mixed crevice and uncreviced potential). However, upon propagation, the more severe crevice corrosion due to the ratio effect, decreased the propagation potential to -500 mV. In contrast, minimum incubation periods to initiation and steady-state propagation varied considerably with solution parameters, and the overall crevice corrosion severity varied accordingly.

Grade Composition Effect:

Grade 1 and 2 consistently varied by approximately one day in minimum incubation periods to initiation. The difference in crevice corrosion severity after a 30 day test was not always detectable, thus it is not surprising that these grades are considered to have similar crevice corrosion resistance. It is apparent, therefore, that potential monitoring provides for a more subtle difference in crevice corrosion resistance.

The somewhat better crevice corrosion resistance of grade 2 is believed to be due to its higher iron content, .10-.15 wt.%, in comparison to 0.02-0.04 wt.% for grade 1. It is possible that minute amounts of ferric ions are present in the crevice liquid boundary layer, during the early stages of corrosion, thus enhancing passivation. Concentrations of 100 ppm ferric ions (Fe^{+3}) have shown to significantly reduce the general corrosion rate of unalloyed titanium in HCl solutions. Such high concentrations of ferric ions are probably not attained in the crevice, but significant concentrations are possible considering the limited crevice volume.

Oxidizers and Crevice/Uncreviced Ratio Effects:

Data on the effect of oxygen concentration on the crevice corrosion of unalloyed titanium are inconsistent, with some studies reporting an increase in susceptibility with increasing concentration^{5,6} and other reporting the opposite^{7,8}. This investigation clearly defined the increase in susceptibility with increasing oxygen concentration, by showing a repeated acceleration of initiation and propagation incubation periods. Overall, the effect of 1000 ppm Fe^{+3} in the bulk solution was similar to that of higher oxygen concentration in accelerating crevice corrosion. These cationic oxidizers act as cathodic depolarizers, and thus accelerate cathodic reduction kinetics, which in turn increases the anodic crevice reaction. These oxidizers will not diffuse into the positively charged active crevice to effect passivation due to similar charge repulsion. On the other hand, certain anionic oxidizing species, such as ClO_3^- , CrO_3^{-2} , and others can migrate into the anodic crevice due to dissimilar charge attraction and inhibit crevice attack.

A crevice (anode) to uncreviced (cathode) surface area ratio larger than or equal to 1:3 had a more pronounced effect on accelerating crevice corrosion than was observed by the addition of oxidizers to a 4:1 ratio. The value of the galvanic current flowing from the anodic crevice is determined by the balance between the anodic reaction in the crevice and cathodic depolarization of the external uncreviced titanium surface. The couple potential at any time is limited by the degree of the cathodic depolarization kinetics as mentioned before, which would depend on oxygen/oxidizer concentration, uncreviced surface area, and solution conductivity at the temperature of interest. Thus, the mechanism of accelerated crevice corrosion is similar for both a larger uncreviced to crevice surface area and the addition of oxygen/oxidizers in their effect on the controlling cathodic depolarization kinetics of the external uncreviced titanium surface. It is important to note that at any ratio of crevice/uncreviced greater than or equal to 1:3, the crevice corrosion accelerating effect was similar in a 5 wt.% NaCl, pH 2, 93°C solution. Thus, at this ratio and solution, the maximum degree of corrosion current was produced.

Alloyed Titanium

Corrosion Behavior:

Alloyed titanium has shown remarkable repassivation cycling, which has not been reported in the literature previously, after crevice initiated, with some decrease in cycling amplitude as propagation approached steady state. Repassivation cycling could be attributed to certain alloying elements such as molybdenum, chromium and vanadium. These elements detected in the crevice corrosion products are believed to be present in the crevice liquid boundary layer as oxidizing species, enhancing passivation. Concentrations of 100 ppm Mo^{+6} , Cr^{+6} , and V^{+5} ions have shown to significantly reduce the general corrosion rate of unalloyed titanium in HCl solutions. Since these elements inhibiting effect is temporary, it is believed that their oxidation states are

lower, than otherwise will be in the bulk solution. More research, beyond the scope of this study, is necessary to conclusively establish their role.

Grade 12 and Ti-3Al-8V-6Cr-4Mo-4Zr Annealed Condition:

Shorter minimum incubation periods to initiation for grade 12, in comparison to Ti-3Al-8V-6Cr-4Mo-4Zr, established their relative crevice corrosion resistance. This relative resistance is in agreement with conventional testing¹¹ who demonstrated a lower pH crevice corrosion resistance for Ti-3Al-8V-6Cr-4Mo-4Zr.

Grade 7 and Beta-21S (Ti-15Mo-2.7Nb-3Al-0.2Si) Annealed Condition:

Beta-21S and grade 7 exhibited a comparably superior crevice corrosion resistance in pH levels as low as 0.2, and 98°C. However, Beta-21S was slower to passivate in comparison to grade 7. Beta-21S also exhibited some repassivation cycling, possibly due to molybdenum enrichment of a less passive surface in comparison to the more noble grade 7.

Grade 2 With Pd Variations 0.01 - 0.05 wt.%

These were experimental compositions and proved to have superior crevice corrosion resistance. The addition of various amounts of palladium to titanium grade 1 and 2 were studied previously^{9,10}. However, at the time, testing was performed in highly concentrated sulfuric and hydrochloric acids, and thus the optimum corrosion resistance was found to be at approximately 2 wt.% palladium. It was noted, presently, that even very low palladium additions provided complete immunity to crevice corrosion at pH levels as low as 0.5 and 95°C. Thus, applications requiring the crevice corrosion resistance of grade 7 that were economically impractical previously, are now feasible using a low Pd alloy. For example, applications involving nuclear waste canister material, where the localized corrosion resistance of grade 7, would be of benefit were not practical due to high cost. Overall, these alloys exhibited a less noble behavior than grade 7, and remained at an active potential at pH 0.5, although crevice corrosion did not occur.

As mentioned previously, higher palladium additions provided for a more noble crevice corrosion behavior. It is believed that the presence of palladium on the surface creates a bi-electrode or galvanic couple on the surface (Pd/matrix), with subsequent anodic polarization of titanium to a noble potential. This assumption is consistent with Stern¹² and Wissenberg¹³ who demonstrated that significant quantity of corroding titanium palladium in HCl solutions did not influence the general corrosion rate of unalloyed titanium. In addition, it was noted that the corrosion rate of titanium palladium was not influenced significantly by the ratio of surface area of metal to volume of corrodent.

Nickel Alloys

Corrosion Behavior:

As mentioned previously, a significant potential drop was not observed upon the onset of crevice corrosion. Thus, potential monitoring technique cannot be effectively utilized to study the crevice corrosion behavior of these alloys. These alloys exhibited an active corrosion potential (E_{corr}) that was very similar to the crevice corrosion potential, and therefore the onset of crevice corrosion was undetectable.

Alloy 625, and C-276 Annealed Condition:

Alloy C-276 was more resistant to crevice attack than Alloy 625, in that Alloy C-276 did not crevice corrode at pH 2. The extent of crevice corrosion of Alloy 625, observed after 30 days was similar to that of titanium grade 2 at pH 2. On the other hand, the extent of corrosion attack of Alloy C-276 was comparable to that of titanium grade 12 and Ti-3Al-8V-6Cr-4Mo-4Zr observed after 30 days at pH 1. The better crevice corrosion resistance of C-276 in comparison to 625 and titanium grades 1 and 2 was also noted by J. Postlethwaite¹⁴ in NaCl solutions 0.1 wt% to saturation, at temperatures ranging from 100° C to 200° C.

SUMMARY

Critical crevice potentials recorded for unalloyed titanium creviced specimens remained generally constant regardless of solution pH, and crevice/uncreviced surface area ratio variations. However, the addition of potent oxidizers such as ferric chloride increased critical potentials significantly.

Alloyed titanium has shown repassivation cycling after crevice corrosion initiated. Critical crevice potentials remained generally constant with solution pH variations.

The onset of crevice corrosion could not be detected in the nickel base alloys tested, using this potential monitoring technique.

Table 12 provides a summary of all the results described for titanium and nickel alloys.

CONCLUSIONS

The research performed during this laboratory examination has led to the following conclusions:

Titanium

1. The potential monitoring technique developed during this program provided a quantitative, real-time signal for monitoring crevice corrosion. This technique was well suited for laboratory applications, and should be suitable for field applications as well.
2. It was feasible to correlate potential measured quantitatively with severity of attack, indicating initiation and propagation of crevice corrosion. Initiation data, alone, provided satisfactory results, and thus a shorter test period was required.
3. This technique distinguished between subtle differences in crevice corrosion resistance.
4. Addition of oxidizers, and change in crevice/uncreviced ratios, was adequately reflected by changes in electrode potential. Thus, shifts from the corrosion potential (E_{corr}) in the active and noble directions denoted increasing and decreasing crevice corrosion attack, respectively.
5. The alloyed titanium tested exhibited remarkable repassivation cycling after crevice corrosion initiated. This phenomena, not reported previously, will require further research.
6. The newly developed titanium alloy, Beta-21S, has proven to possess superior crevice corrosion resistance to pH levels as low as 0.2.
7. The superior crevice corrosion resistance of grade 2 with small palladium additions (0.01-0.05 wt.%), observed at pH levels as low as 0.5, should possibly provide a candidate material for nuclear waste canisters. Thus, applications requiring the localized corrosion resistance of grade 7 that were economically impractical previously, are now feasible.

Nickel Alloys

1. The nickel based alloys tested exhibited an active corrosion potential (E_{corr}) that was near the crevice corrosion potential. Therefore, the onset of crevice corrosion was not detected in these alloys.
2. The crevice corrosion resistance of alloy 625 was similar to that of titanium grade 2, in that both exhibited moderate attack at pH 2 and 93°C. The crevice corrosion resistance of Alloy C-276 was comparable to that of titanium grade 12 and Ti-3Al-8V-6Cr-4Mo-4Zr.

Future Work

It is concluded that potential monitoring technique offers promise as an on-line device for early detection and monitoring of crevice corrosion. Plans are underway to design a full scale prototype, and to conduct field trials in operating heat exchangers.

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Table 1

Nominal Composition of Unalloyed Titanium

ASTM Grade 1: 0.06O, 0.02Fe, 0.005H, 0.01C, 0.006N
(alpha)

ASTM Grade 2: 0.10O, 0.15Fe, 0.005H, 0.01C, 0.006N
(alpha)

Composition of Alloyed Titanium

ASTM Grade 12: 0.12O, 0.13Fe, 0.005H, 0.01C, 0.006N,
(near-alpha) 0.3Mo, 0.7Ni

ASTM Grade 7: 0.10O, 0.15Fe, 0.005H, 0.01C, 0.006N,
(alpha) 0.12Pd

Beta-C: 3.5Al-4.0Mo-8.0V-4.0Zr-6.0Cr-0.09O-< 0.03Fe
(beta)

Beta-21S: 3Al, 15Mo, 2.7Nb, 0.2Si, 0.13O, 0.15Fe
(beta)

Composition of Nickel Alloys

Inco Alloy C-276: 58Ni, 5Fe, 16Cr, 16Mo, 4W, 0.35V

Inconel Alloy 625: 61Ni, 2.5Fe, 21.5Cr, 9Mo, 3.6Ta

Table 2(a)

Crevice Corrosion Grade 1 and 2

Potential (mV) vs. Ag/AgCl

5% NaCl, pH 2, 93°C

Creviced Specimens

Grade 1

<u>Day</u>	<u>(1)</u>	<u>(2)</u>	<u>(3)</u>	<u>(4)</u>	<u>(5)</u>	<u>(6)</u>
1 (24 Hrs)	-223	-131	-200	-255	-112	-269
2	-288	-170	-260	-300	-180	-285
3	-300	-190	-295	-360	-280	-347
4	-340	-250	-350		-327	-360
5	-368	-300	-370		-380	-378
6	-380	-340	-385		-390	-380
9	-400	-390	-420		-410	-405

Grade 2

<u>Day</u>	<u>(1)</u>	<u>(2)</u>	<u>(3)</u>	<u>(4)</u>	<u>(5)</u>	<u>(6)</u>
1	-160	- 80	-240	-200	-168	-180
2	-186	-178	-285	-265	-190	-230
3	-280	-265	-305	-296	-250	-286
4	-300	-285	-330	-320	-290	-325
5	-340	-326	-380	-360	-330	-350
6	-370	-349	-400	-385	-390	-380
9	-420	-378	-430	-400	-430	-412

Table 2(a) (Continued)

Crevice Corrosion Grade 1 and 2

Potential (mV) vs. Ag/AgCl

5% NaCl, pH 2, 93°C

Creviced Specimens

Grade 1

<u>Day</u>	<u>(7)</u>	<u>(8)</u>	<u>(9)</u>	<u>(10)</u>
1	-260	-131	-223	-255
2	-284	-250	-262	-300
3	-300	-320	-285	
4	-365		-345	
5			-365	
8			-370	
9			-394	
15			-470	
16			-460	
17			-440	
21			-475	
22			-480	
26			-480	
27			-490	
28			-460	
29			-455	
30			-440	

Grade 2

<u>Day</u>	<u>(7)</u>	<u>(8)</u>	<u>(9)</u>	<u>(10)</u>
1	- 90	-246	-230	-240
2	-150	-260	-300	-316
3	-200	-300	-320	
4	-265	-320	-345	
5	-300	-400		
8	-347	-430		
9	-358	-422		
15	-400	-426		
16	-420	-440		
17	-433	-450		
21	-480	-460		
22	-460	-465		
26	-467	-460		
27	-435	-440		
28	-430	-443		
29	-445	-440		

Table 2(b)

Crevice Corrosion Grade 1 and 2

Average Potential of Five Specimens (mV) vs. Ag/AgCl

5% NaCl, pH 2, 93°C

Creviced Specimens

<u>Day</u>	<u>Gr 1</u>	<u>Gr 2</u>	<u>Gr 1 Free</u>	<u>Gr 2 Free</u>
0	-250	-240	-130	-120
1	-220	-240	+180	+184
2	-280	-320	+340	+341
3	-320	-330	+410	+375
4	-360	-340	+415	+410
5	-365	-400	+410	+410
8	-375	-430		
9	-392	-422		
15	-460	-433		
16	-456	-420		
17	-450	-430		
21	-485	-480		
22	-470	-460		
26	-440	-465		
27	-480	-430		
28	-485	-430		
29	-480	-438		
30				

Table 3

Crevice Corrosion Grade 1 and 2

Average Potential of Four Specimens (mV) vs. Ag/AgCl

5% NaCl, pH 2, 93°C, Air Sparged

Creviced Specimens

<u>Day</u>	<u>Gr 1</u>	<u>Gr 2</u>	<u>Gr 1 Free</u>	<u>Gr 2 Free</u>
0	-280	-250	+470	+420
1	-330	-260	+460	+430
2	-390	-330		
3	-396	-360		
5	-380	-410	+465	+437
6	-440	-485		
7	-420	-470		
8	-460	-450		
30	-487	-490		

Table 4

Crevice Corrosion Grade 1 and 2

Average Potential of Three Specimens (mV) vs. Ag/AgCl

5% NaCl, pH 2, 1000 ppm Fe 93°C
With 1000 ppm Fe Inside the Crevice

Crevice Specimens

<u>Day</u>	<u>Gr 1</u>	<u>Gr 2</u>	<u>Gr 1 Free</u>	<u>Gr 2 Free</u>
0	+350	+380	+400	+390
1	+613	+607	+630	+632
2	+612	+620	+621	+620
3	+608	+608	+620	+620
4	+600	+600	+608	+608
5	+595	+595	+598	+598
9	+583	+584	+586	+587
10	+576	+578	+578	+579
12	+564	+566	+568	+567
30	+550	+556	+570	+574

Without 1000 ppm Fe Inside Crevice

<u>Day</u>	<u>Gr 1</u>	<u>Gr 2</u>	<u>Gr 1 Free</u>	<u>Gr 2 Free</u>
0	+323	+330	+400	+400
1	+548	+530	+630	+620
2	+515	+525	+621	+625
3	+500	+505	+620	+625
4	+460	+475	+620	+620
5	+370	+365	+605	+610
6	+310	+300		
7	+300	+310	+580	+590
30	+290	+298	+570	+580

Table 5

Crevice Corrosion Grade 1 and 2

Average Potential of Three Specimens (mV) vs. Ag/AgCl

5% NaCl, pH 5, 93°C

Crevice Specimens

<u>Day</u>	<u>Gr 1</u>	<u>Gr 2</u>	<u>Gr 1 Free</u>	<u>Gr 2 Free</u>
0	-250	-230	-120	-115
1	+ 55	+ 42	+200	+220
2	-230	- 62	+350	+360
3	-264	- 70	+420	+400
6	-360	-212	+420	+415
7		-352	+422	+413
10	-380	-370		
12	-385	-390		
15	-400	-405		
17	-448	-420		
30	-460	-450		

Table 6

Crevice Corrosion Grade 1

Potential (mV) vs. Ag/AgCl
5% NaCl, pH 2, 93°C

Crevice/Uncreviced Ratio 1:3

<u>Day</u>	<u>1</u>	<u>2</u>	<u>3</u>
1	-320	-340	-335
2	-340		-360
3	-500		-430
4	-480		-485
5	-470		-488
6	-490		-490
15	-465		-480
17	-487		-480
30	-480		-480

Table 7

Crevice Corrosion Grade 1

Potential (mV) vs. Ag/AgCl
5% NaCl, pH 2, 93°C

Crevice/Uncreviced Ratio 1:90

<u>Day</u>	<u>1</u>	<u>2</u>	<u>3</u>
1	-300	-305	-300
2		-150	-135
3		-260	-265
4			-270
5			-290
6			-400
15			-456
30			-465

Table 8(a)

Crevice Corrosion Grade 12 and 7

Average Potential of Three Specimens (mV) vs. Ag/AgCl

5% NaCl, pH 1, 95°C

<u>Day</u>	<u>Gr 12</u>	<u>Gr 7</u>	<u>Gr 12 Free</u>	<u>Gr 7 Free</u>
0	-115	-124	-161	-124
1	- 82	+105	+250	+293
2	- 87	+163	+270	+345
3	- 83	+188	+373	+467
4	- 94	+300	+390	+470
5	+101	+388	+395	+470
6		+378		+460
7	- 90	+393		+450
8	-150	+401		+455
9	+ 30	+400	+455	
10	-165			
12	+ 10			
13	-170			
14	- 65	+425		
15	-168			
17	- 70	+420		

Table 8(b)

5% NaCl, pH 0.5, 95°C

<u>Day</u>	<u>Gr 12</u>	<u>Gr 7</u>	<u>Gr 12 Free</u>	<u>Gr 7 Free</u>
0	-245	-224	-200	-190
1	- 80	+ 5	+240	+280
2	- 90	+ 97		
3	-100	+130		
4	-130	+164	+380	+460
5	-152	+186		
6	+ 55	+210		
9	- 61	+217	+400	+430
10	- 80	+217	+570	
11	-142	+214		
12	- 55	+337	+395	+420
13	-175			
14	- 90			
15	-180	+350		

Table 9(a)

Crevice Corrosion Grade 2 Alloyed With 0.01, 0.02, 0.05 Wt.% Pd

Average Potential (mV) of Three Specimens
vs. Ag/AgCl 5% NaCl, pH 1, 95°C

<u>Day</u>	<u>Gr. 2</u> <u>(0.01 Wt.%)</u>	<u>Gr. 2</u> <u>(0.02 Wt.%)</u>	<u>Gr. 2</u> <u>(0.05 Wt.%)</u>	<u>Gr. 7</u>
1	- 30	+ 44	+ 86	+105
2	-260	-220	-170	+163
4	+200	+230	+280	+300
5	+280	+285	+280	+388
7	+340	+350	+365	+393
9	+300	+282	+350	+400
10	+200	+220	+240	
12	+180	+180	+210	
15	+185	+187	+200	
17	+185	+190	+220	
19	+200	+205	+220	+416
24	+228	+230	+284	
30	+220	+249	+290	+425

Table 9(b)

Crevice Corrosion Grade 2 Alloyed With 0.01, 0.02, 0.05 Wt.% Pd

Average Potential (mV) of Three Specimens
vs. Ag/AgCl 5% NaCl, pH 0.5, 95°C

<u>Day</u>	<u>Gr. 2</u> <u>(0.01 Wt.%)</u>	<u>Gr. 2</u> <u>(0.02 Wt.%)</u>	<u>Gr. 2</u> <u>(0.05 Wt.%)</u>	<u>Gr. 7</u>
1	-220	-210	-218	+ 5
2	-240	-220	-225	+ 97
3	-245	-225	-200	-130
4	-260	-252	-220	-164
5	-260	-257	-230	-186
6	-273	-250	-226	-210
7	-280	-260	-240	
8	-283	-270	-255	
11	-280	-278	-260	+214
21	-300	-290	-293	
30	-310	-296	-290	

Table 10(a)

Crevice Corrosion Beta-21S and Beta-C

Average Potential (mV) of Three Specimens vs. Ag/AgCl
5% NaCl, pH 1, 95°C

<u>Day</u>	<u>Beta-21S</u>	<u>Beta C</u>	<u>Beta-21S Free</u>	<u>Beta C Free</u>
0	-223	-258	-196	-162
1	-140	- 92	+341	+340
2	- 87	-174	+370	+395
3	- 80	-140	+404	+453
4	- 75	-145	+437	+458
5	- 63	-157	+437	+463
6	+ 48	-150	+471	+480
7	+ 0.3	-105	+480	+494
8	+ 16.5	+166.3		
12	+ 50.2	- 7.3	+480	+490
13	+ 13.0	-270		
15	+ 5.8	-304		
16	+ 22.1	+ 60		
19	- 91.3	-300		
20	- 73	+ 40	+470	+465
21	- 78	-320		
22		-300		
25	- 60	- 60		
27		-330		
30	- 50	- 80		

Table 10(b)

Crevice Corrosion Beta-21S and Beta-C

Average Potential (mV) of Three Specimens vs. Ag/AgCl
5% NaCl, pH 0.5, 95°C

<u>Day</u>	<u>Beta-21S</u>	<u>Beta C</u>	<u>Beta-21S Free</u>	<u>Beta C Free</u>
0	-250	-300	-220	-180
1	-132	-168	+346	+340
2	- 84	-137	+360	+370
3	- 78	-177	+400	+430
4	- 82	- 25	+420	+440
5	- 69	-370	+430	+455
6	- 32	-264	+460	+480
9	- 20	-273		
10	- 25	+ 73	+440	+456
11	- 38.5	-340		
13	- 10.9	-200		
17	+ 73	+ 20		
18	+ 88.6	-300		
20		-360		
22		-375		
24	+ 90	- 60		
25		-300		
26	+100	-200		
27		-128		
28	+105	-310		

Table 10(c)

Crevice Corrosion Beta-21S

Average Potential (mV) of Three Specimens vs. Ag/AgCl
5% NaCl, pH 0.2, 98°C

<u>Day</u>	<u>Beta-21S</u>	<u>Beta-21S Free</u>
0	-220	-200
1	- 70	- 67
3	- 76	- 55
4	- 76	- 42
6	- 56	
7	- 79	- 53
8	-128	
9	-220	- 44
10	-265	
11	-260	- 58
30	-317	- 35
32	- 55	- 30

Table 11 (a)

Nickel Alloy 625

Averaged Potential (mV) of Three Specimens vs. Ag/AgCl
5% NaCl, pH 2, 95°C

<u>Day</u>	<u>625</u>	<u>625 Free</u>
0	-250	-240
1	-255	-250
2	-260	-250
3	-255	-255
4	-260	-255
5	-260	-255
15	-270	-265
20	-290	-275
30	-270	-260

Table 11 (b)

Nickel Alloys 625 and C-276

Averaged Potential (mV) of Three Specimens vs. Ag/AgCl
5% NaCl, pH 1, 95°C

<u>Day</u>	<u>625</u>	<u>C-276</u>	<u>625 Free</u>	<u>C-276 Free</u>
0	-300	-280	-280	-270
1	-320	-290	-300	-285
2	-330	-300	-320	-290
10	-350	-310	-340	-300
30	-340	-300	-330	-297

Table 11 (c)

Nickel Alloys 625 and C-276

Averaged Potential (mV) of Three Specimens vs. Ag/AgCl
5% NaCl, pH 0.5, 95°C

<u>Day</u>	<u>625</u>	<u>C-276</u>	<u>625 Free</u>	<u>C-276 Free</u>
0	-400	-300	-390	-280
1	-405	-300	-395	-290
2	-410	-305	-400	-300
10	-430	-320	-415	-310
20	-440	-330	-430	-320
30	-430	-330	-420	-325

Table 12

Results Summary

<u>Alloy</u>	<u>Variations of Standard Solution¹</u>	<u>Crevice/Uncreviced Ratio</u>	<u>Initiation: Potential (mV)</u>	<u>Time (Days)</u>	<u>Propagation Potential (mV)</u>	<u>Time (Days)</u>
Ti Grade 1	None	4/1	-320	3	-440 to -485	10
	Air Sparging	4/1	-320	2	-440 to -485	6
	Fe+3 (1000 ppm) ²	4/1	+515	2	+330 to +300	6
	pH 5	4/1	-320	6	-440 to -460	17
	None	1/3	-330	1	-430 to -480	4
	None	1/90	-300	1	-270 to -460	5
Ti Grade 2	None	4/1	-345	4	-440 to -480	10
	Air Sparging	4/1	-345	3	-440 to -485	6
	Fe+3 (1000 ppm) ²	4/1	+505	3	+330 to +300	6
	pH 5	4/1	-345	7	-420 to -450	17
Ti Grade 7	95°C, pH 1	4/1	Did Not Crevice			
	95°C, pH 0.5	4/1	Did Not Crevice			
Ti Grade 12	95°C, pH 1	4/1	-150	8	Repassivation Cycles -150 to +30	1*
	95°C, pH 0.5	4/1	-150	5	Repassivation Cycles -152 to +55	1*
Beta-C	95°C, pH 1	4/1	-304	15	Repassivation Cycles -304 to +60	1*
	95°C, pH 0.5	4/1	-370	5	Repassivation Cycles -370 to +73	5*
Beta-21S	95°C, pH 1	4/1	Did Not Crevice			
	95°C, pH 0.5	4/1	Did Not Crevice			
	98°C, pH 0.2	4/1	Did Not Crevice			

*Between cycles after initiation.

¹Standard Solution: 5% NaCl, 93°C, pH 2, Natural Aeration.

²Crevicees were assembled in standard solution (without Fe+3).

Table 12 (Continued)

Results Summary

<u>Alloy</u>	<u>Variations of Standard Solution¹</u>	<u>Crevice/Uncreviced Ratio</u>	<u>Initiation: Potential (mV)</u>	<u>Time (Days)</u>	<u>Propagation Potential (mV)</u>	<u>Time (Days)</u>
Ti Grade 2 (0.01 wt.% Pd)	95°C, pH 1	4/1	Did Not Crevice			
	95°C, pH 0.5	4/1	Did Not Crevice			
Ti-Grade 2 (0.02 wt.% Pd)	95°C, pH 1	4/1	Did Not Crevice			
	95°C, pH 0.5	4/1	Did Not Crevice			
Ti-Grade 2 (0.05 wt.% Pd)	95°C, pH 1	4/1	Did Not Crevice			
	95°C, pH 0.5	4/1	Did Not Crevice			
C-276	95°C, pH 2	4/1	Did Not Crevice			
	95°C, pH 1	4/1	Crevice Corroded			
	95°C, pH 0.5	4/1	Crevice Corroded			
Alloy 625	95°C, pH 2	4/1	Crevice Corroded			
	95°C, pH 1	4/1	Crevice Corroded			
	95°C, pH 0.5	4/1	Crevice Corroded			

¹Standard Solution: 5% NaCl, 93°C, pH 2, Natural Aeration.

²Crevices were assembled in standard solution (without Fe⁺³).

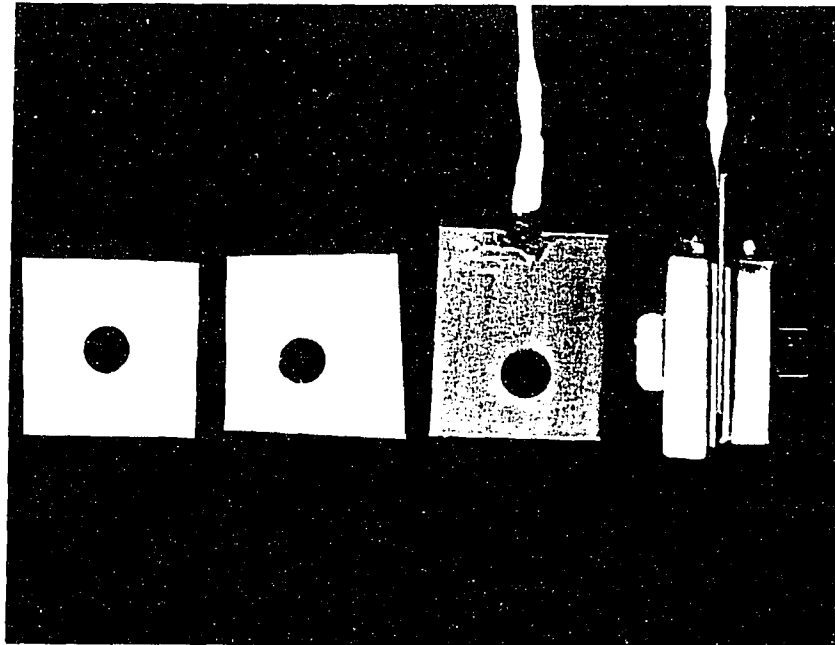


Figure 1. Electrochemical crevice cell.

From left to right: Two Teflon sheets, crevice specimen,
and assembled crevice cell.

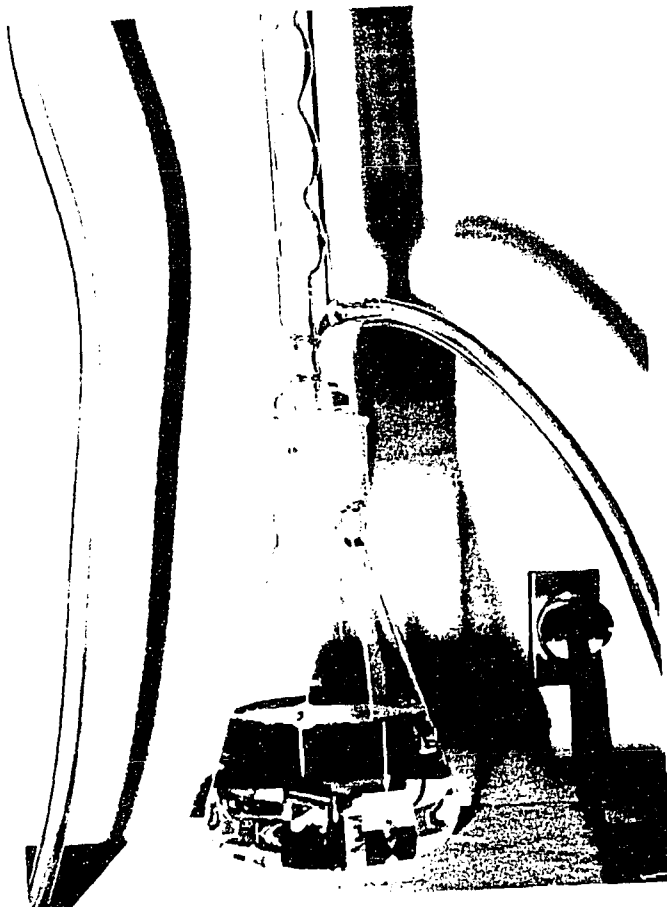


Figure 2. Huey corrosion test apparatus.

Figure 3a

**CREVICE CORROSION POTENTIAL
TITANIUM GRADES 1 AND 2
PH 2**

AVERAGE POTENTIAL (mV) VS. Ag/AgCl REFERENCE

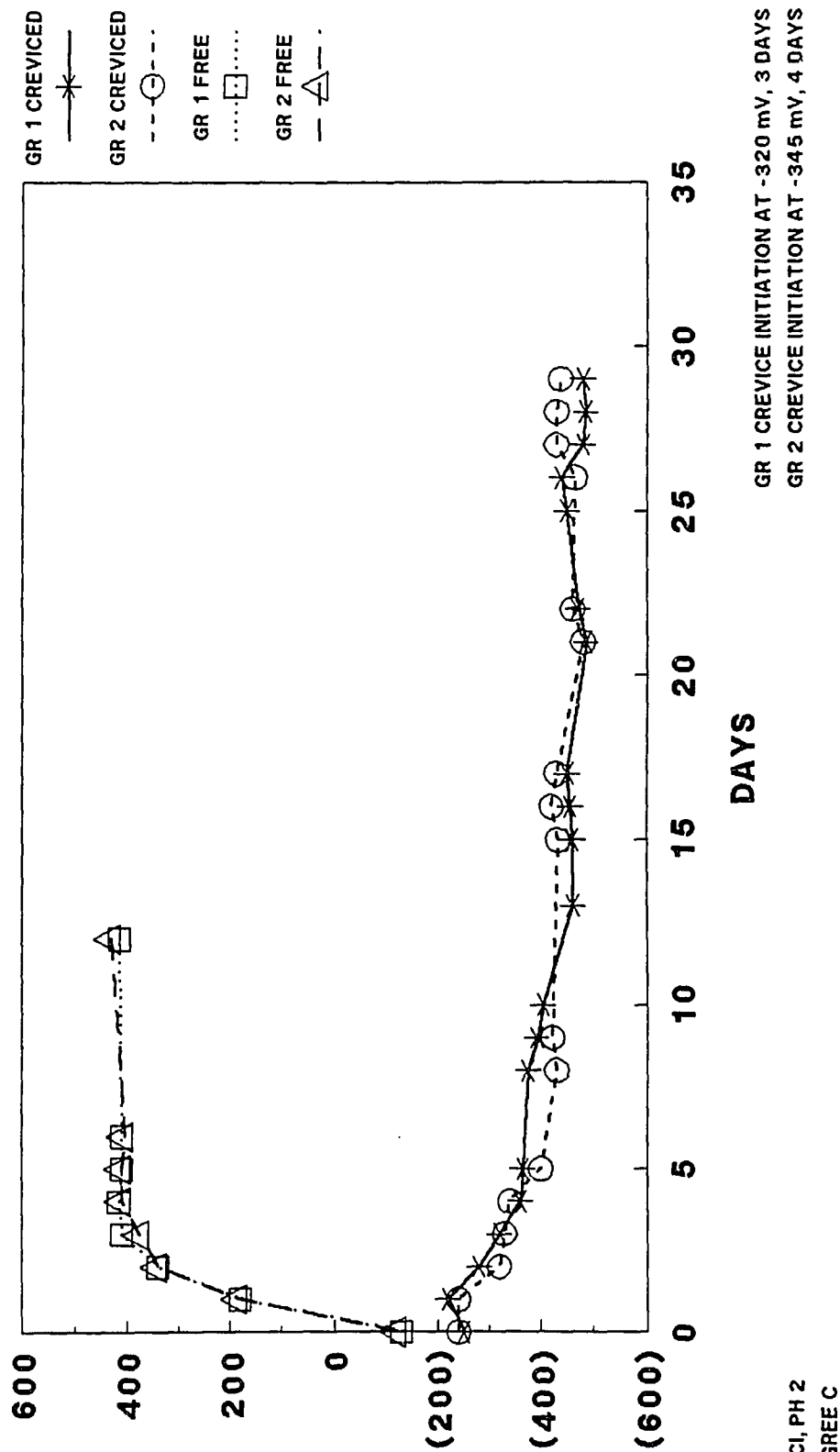


Figure 3b

**CREVICE CORROSION POTENTIAL
TITANIUM GRADES 1 AND 2
PH 2 (5 DAYS)**

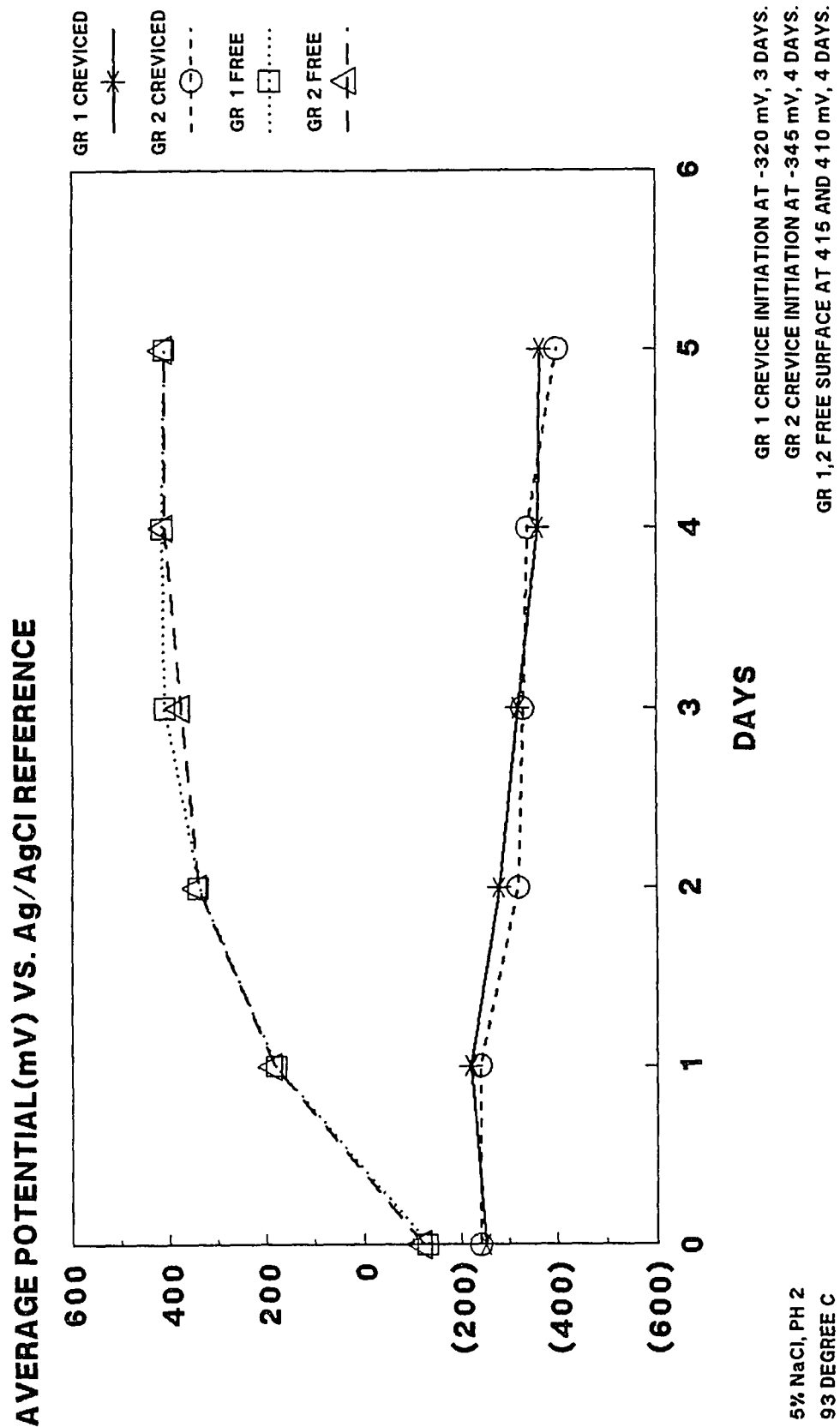


Figure 4

**CREVICE CORROSION POTENTIAL
TITANIUM GRADES 1 AND 2
PH 2, AIR SPARGING (5 DAYS)**

AVERAGE POTENTIAL(mV) VS. Ag/AgCl REFERENCE

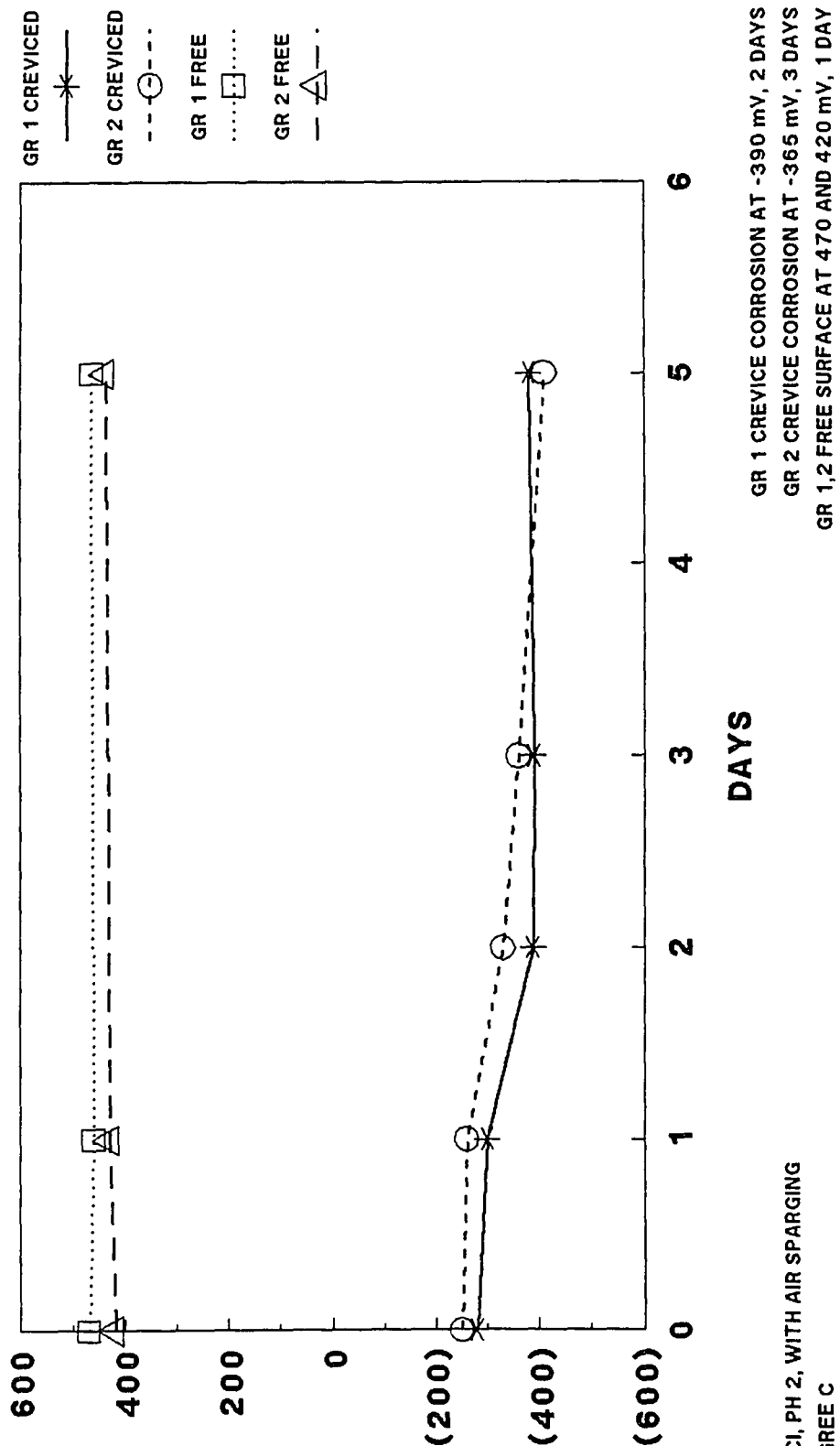
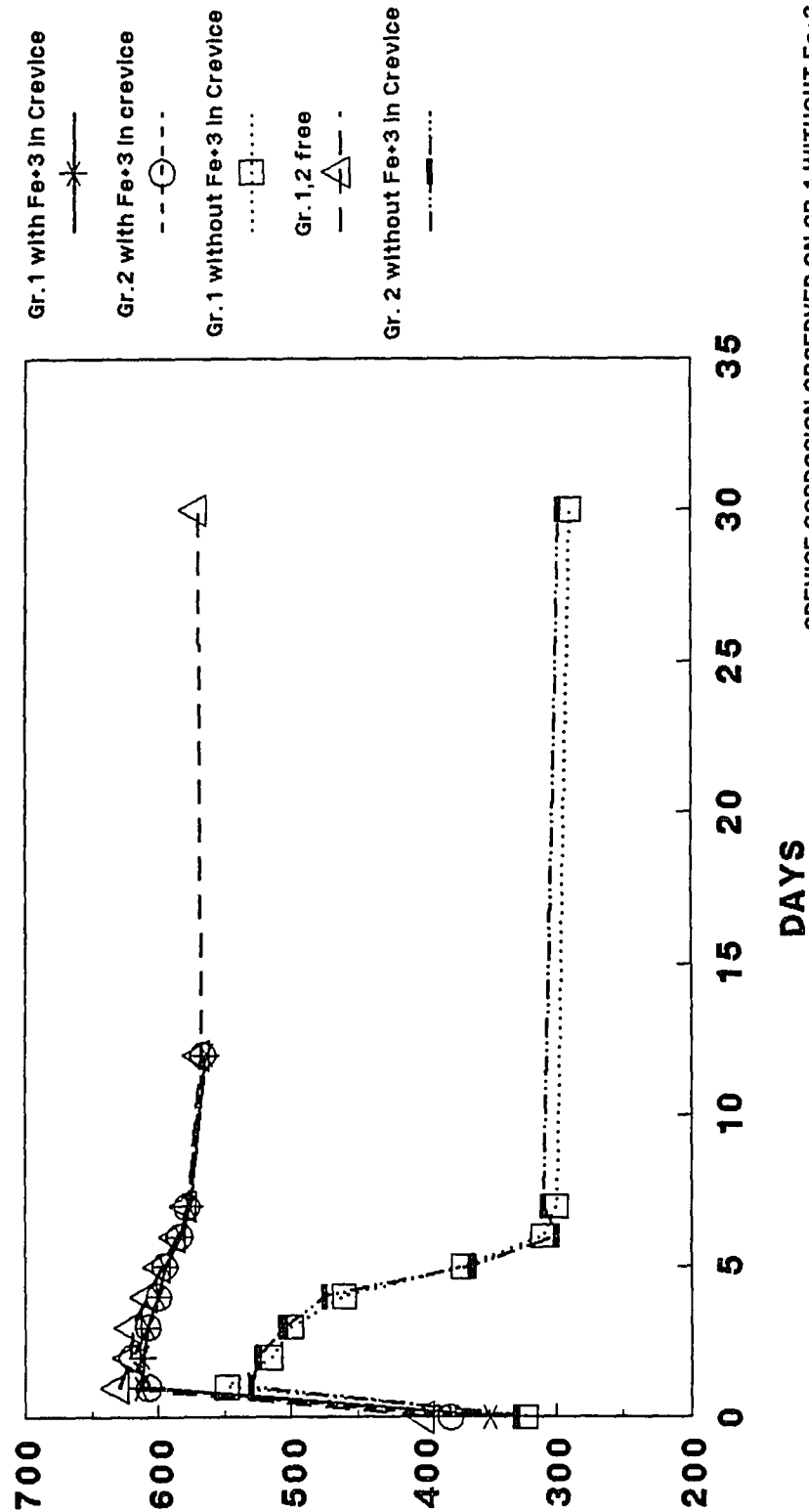


Figure 5

**CREVICE CORROSION POTENTIAL
TITANIUM GRADE 1 AND 2
PH 2, WITH FERRIC CHLORIDE ADDITION**

AVERAGE POTENTIAL (mV) VS. Ag/AgCl REFERENCE



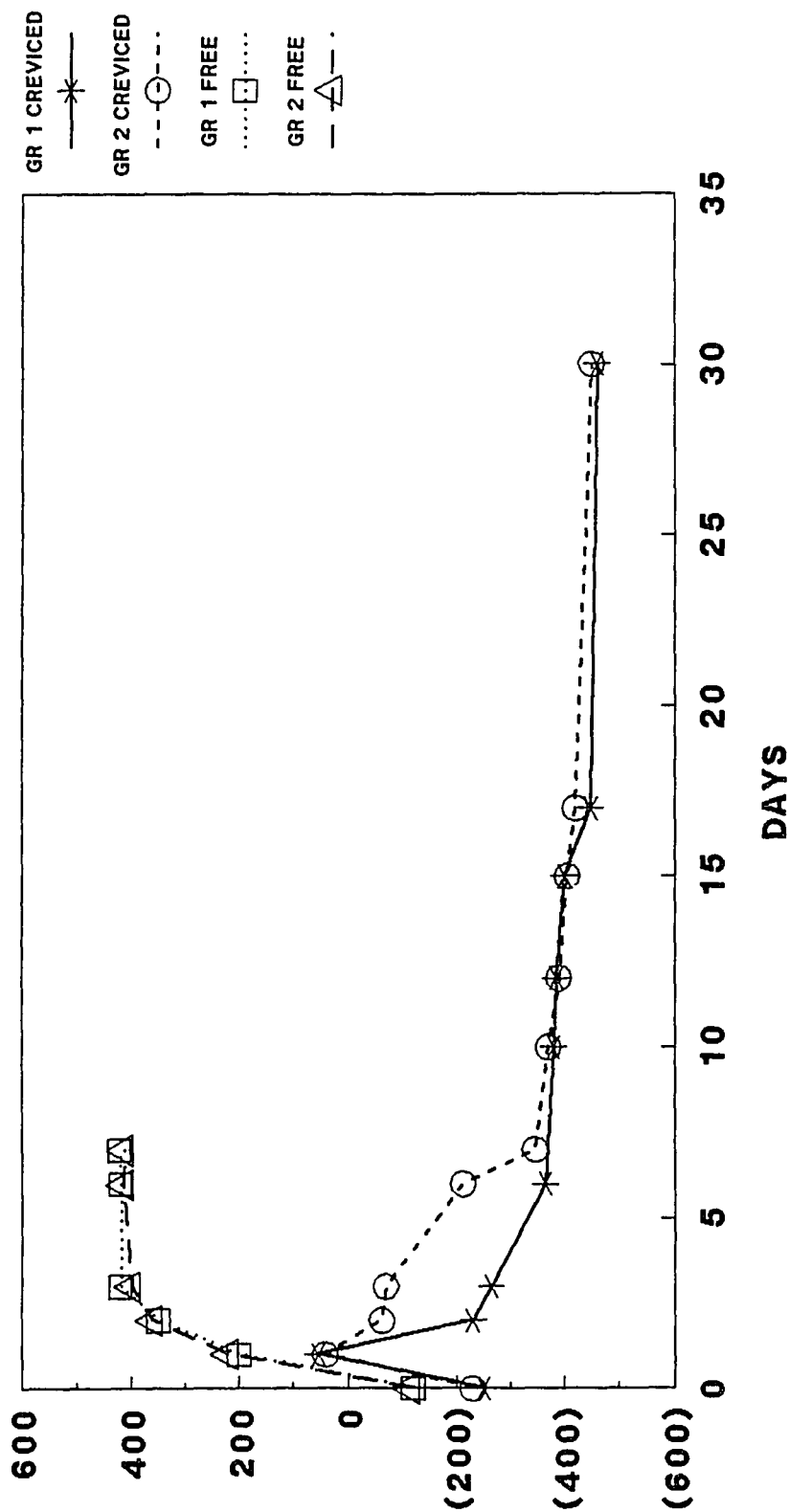
CREVICE CORROSION OBSERVED ON GR. 1 WITHOUT Fe+3
IN CREVICE AT 515 mV, 2 DAYS. CREVICE CORROSION
WAS NOT FOUND ON GR. 1, 2 WITH Fe+3 IN CREVICE.

5% NaCl, WITH 1000 PPM Fe+3, PH 2
93 DEGREE C

Figure 6

**CREVICE CORROSION POTENTIAL
TITANIUM GRADES 1 AND 2
PH 5**

AVERAGE POTENTIAL(mV) VS. Ag/AgCl REFERENCE



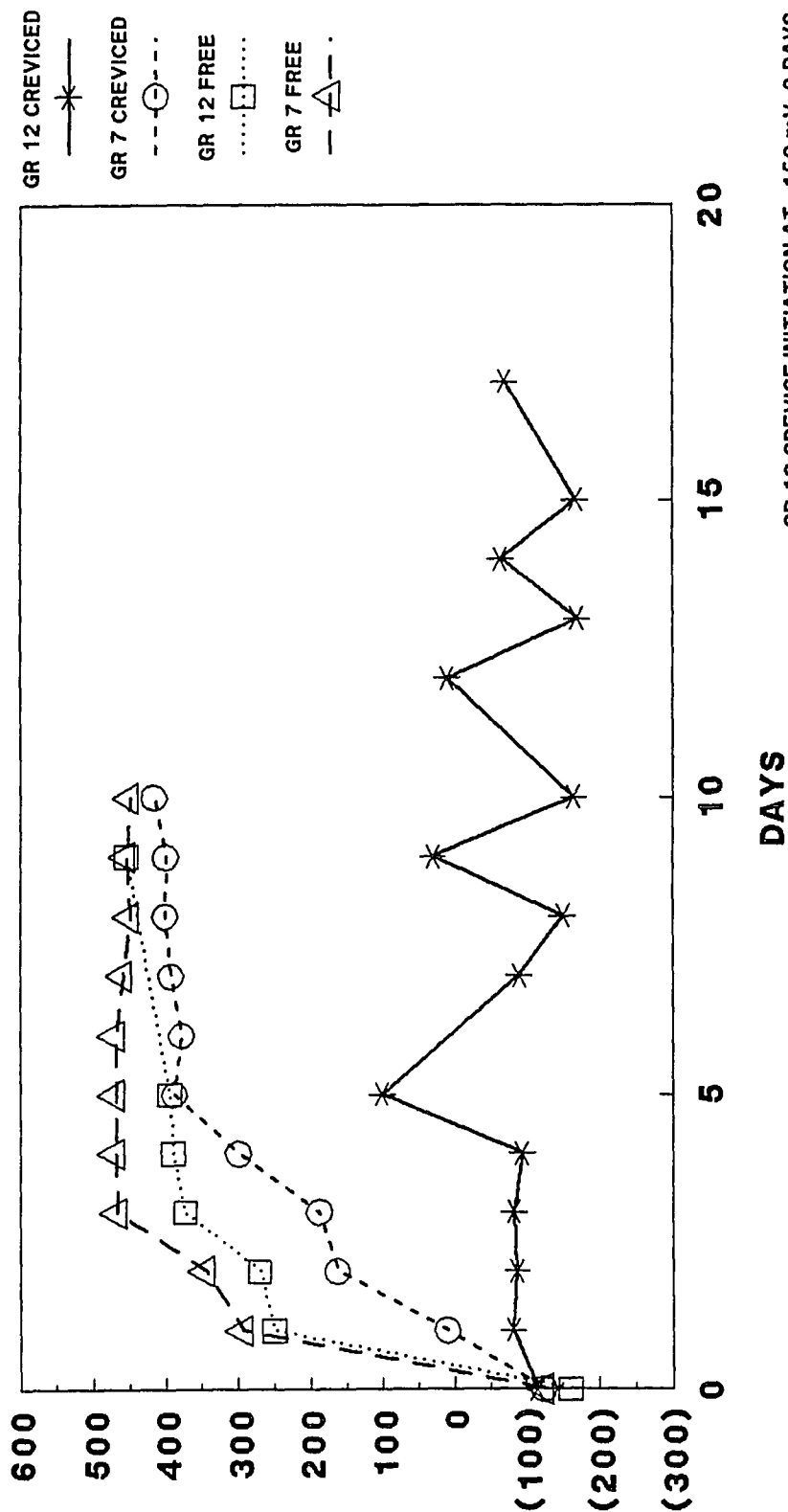
5% NaCl, PH 5
93 DEGREE C

CREVICE CORROSION, GR. 1, AT -365mV, 6 DAYS
CREVICE CORROSION, GR. 2, AT -346mV, 7 DAYS

Figure 7a

**CREVICE CORROSION POTENTIAL
TITANIUM GRADES 12 AND 7
PH 1**

AVERAGE POTENTIAL (mV) VS. Ag/AgCl REFERENCE



GR 12 CREVICE INITIATION AT -150 mV, 8 DAYS.
WHEN CORROSION INITIATED, PARTIAL PASSIVATIONS
WERE ACHIEVED DUE TO CORROSION PRODUCT OXIDIZERS

5% NaCl, PH 1
95 DEGREE C

Figure 7b

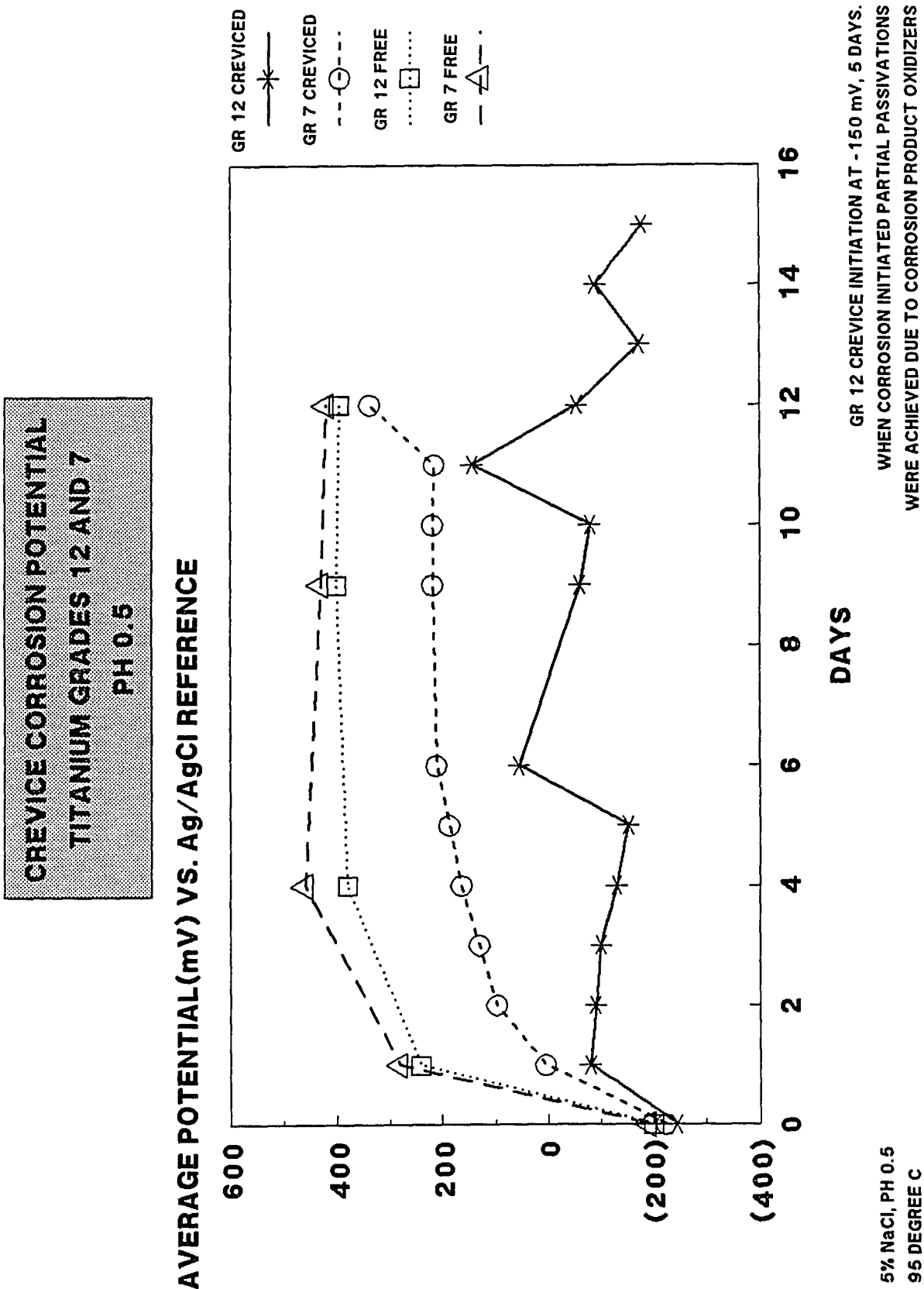
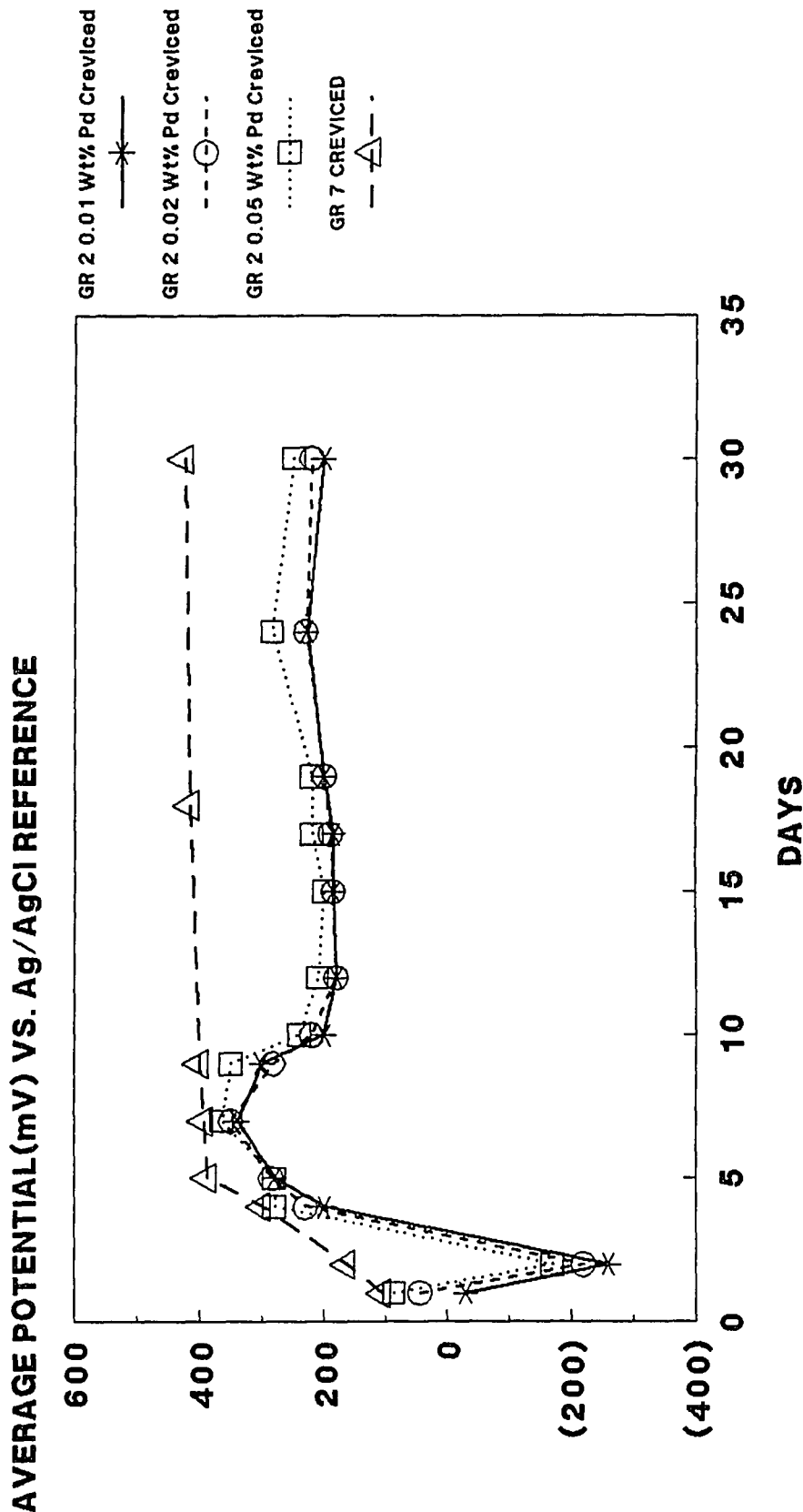


Figure 8a

**CREVICE CORROSION POTENTIAL
TITANIUM GRADES 7 AND 2(Pd) 0.01, 0.02, 0.05 Wt%
PH 1**



5% NaCl, PH 1
95 DEGREE C

CREVICE CORROSION DID NOT OCCUR

Figure 8b

CREVICE CORROSION POTENTIAL

TITANIUM GRADES 7 AND 2(Pd) 0.01, 0.02, 0.05 Wt%

PH 0.5

AVERAGE POTENTIAL(mV) VS. Ag/AgCl REFERENCE

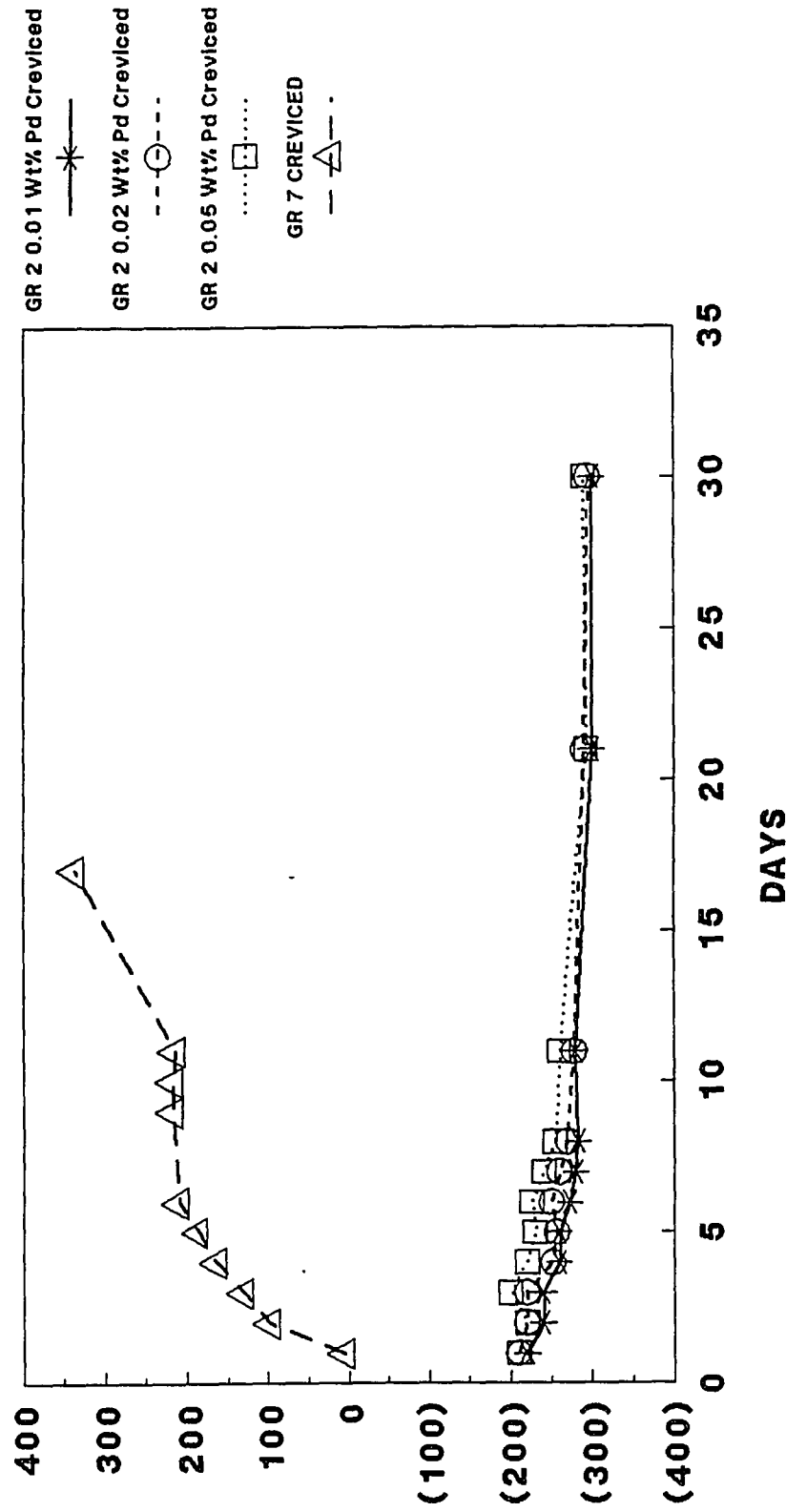
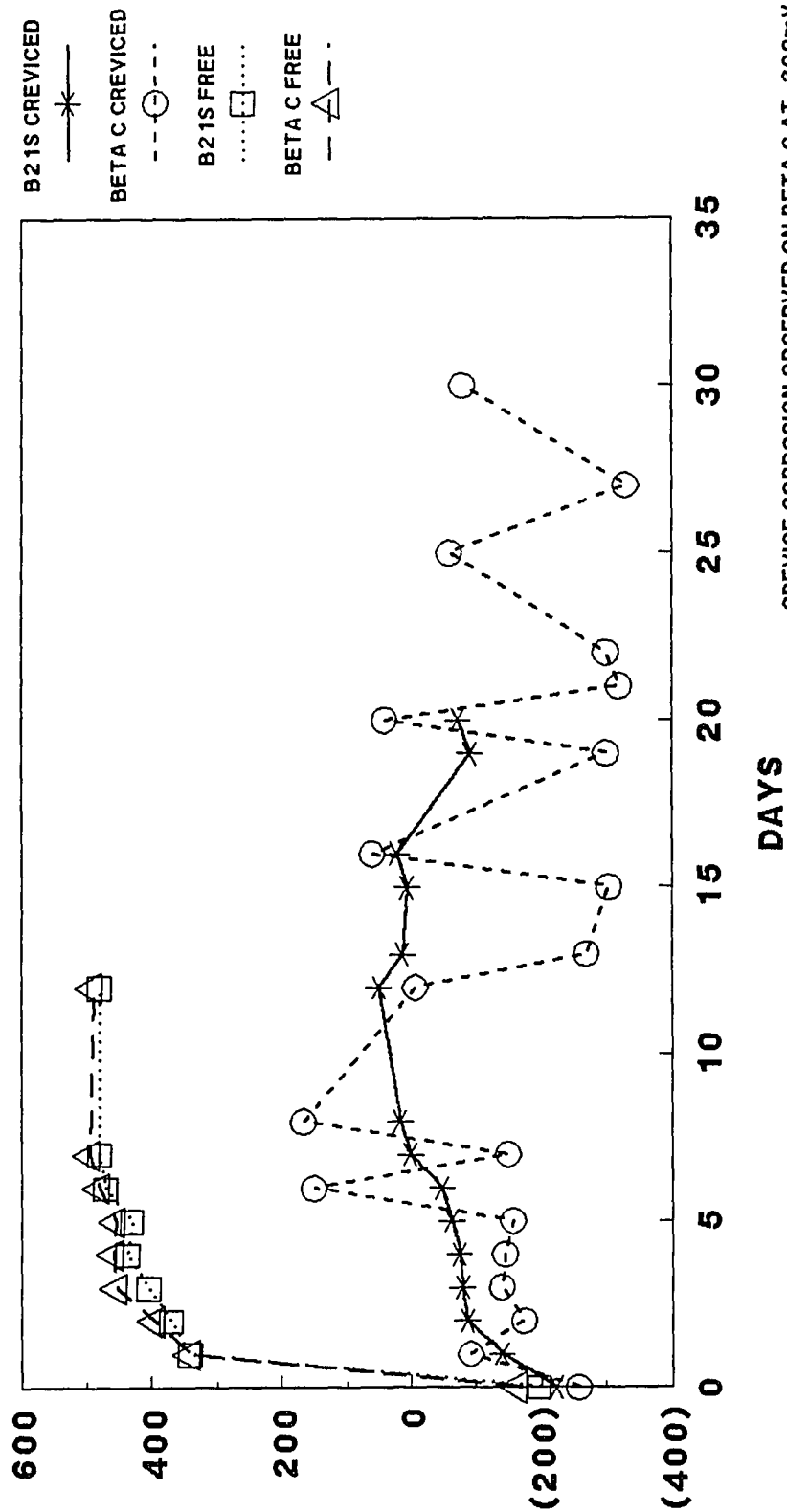


Figure 9a

**CREVICE CORROSION POTENTIAL
TITANIUM BETA-2 1S AND BETA C
PH 1**

AVERAGE POTENTIAL (mV) VS. Ag/AgCl REFERENCE



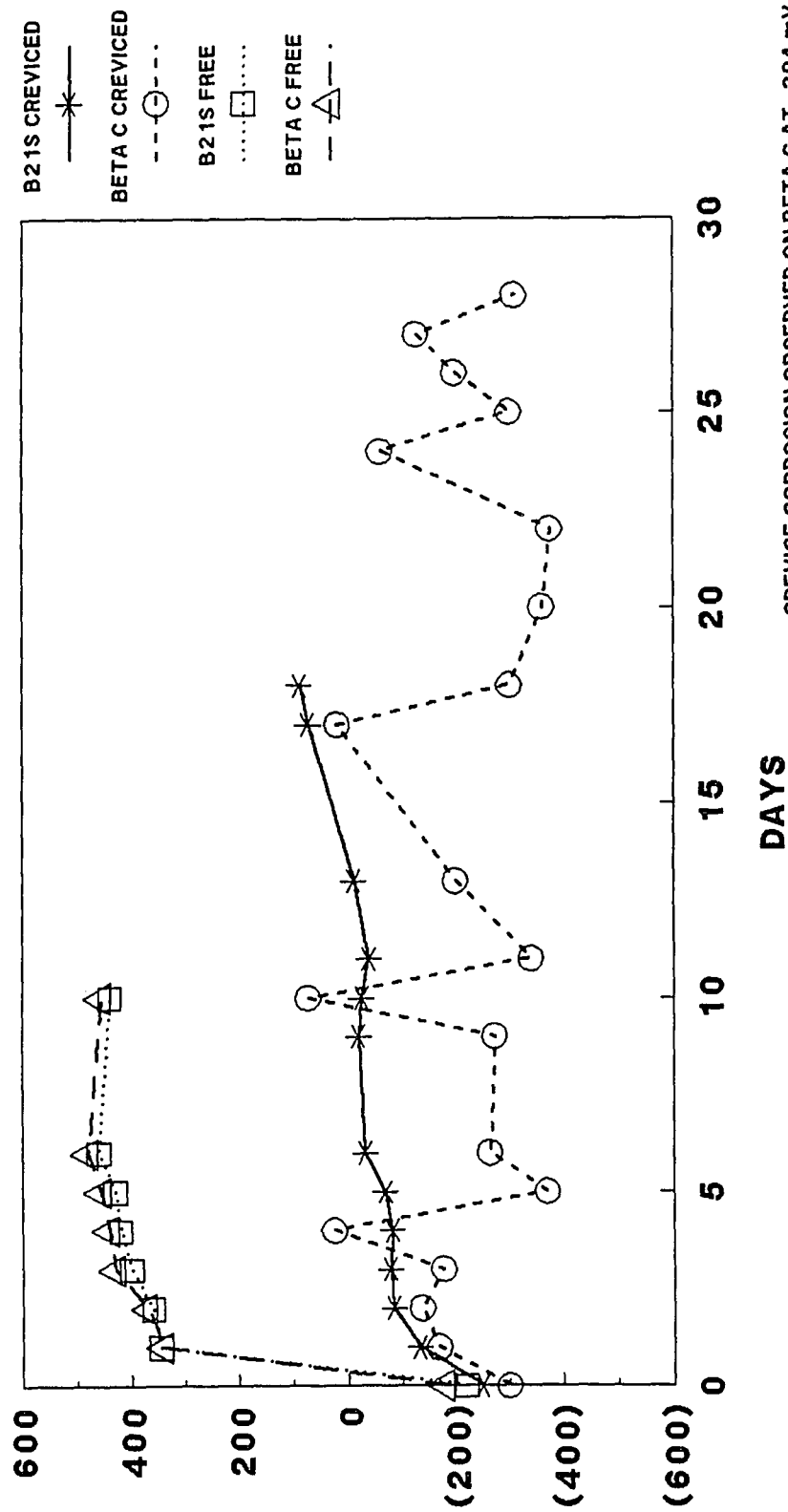
CREVICE CORROSION OBSERVED ON BETA C AT -300mV
AFTER 15 DAYS. WHEN CREVICE INITIATED PARTIAL
PASSIVATIONS WERE DUE TO CORR. PRODUCT OXIDIZERS

5% NaCl, PH 1
95 DEGREE C

Figure 9b

**CREVICE CORROSION POTENTIAL
TITANIUM BETA-2 1S AND BETA C
PH 0.5**

AVERAGE POTENTIAL(mV) VS. Ag/AgCl REFERENCE

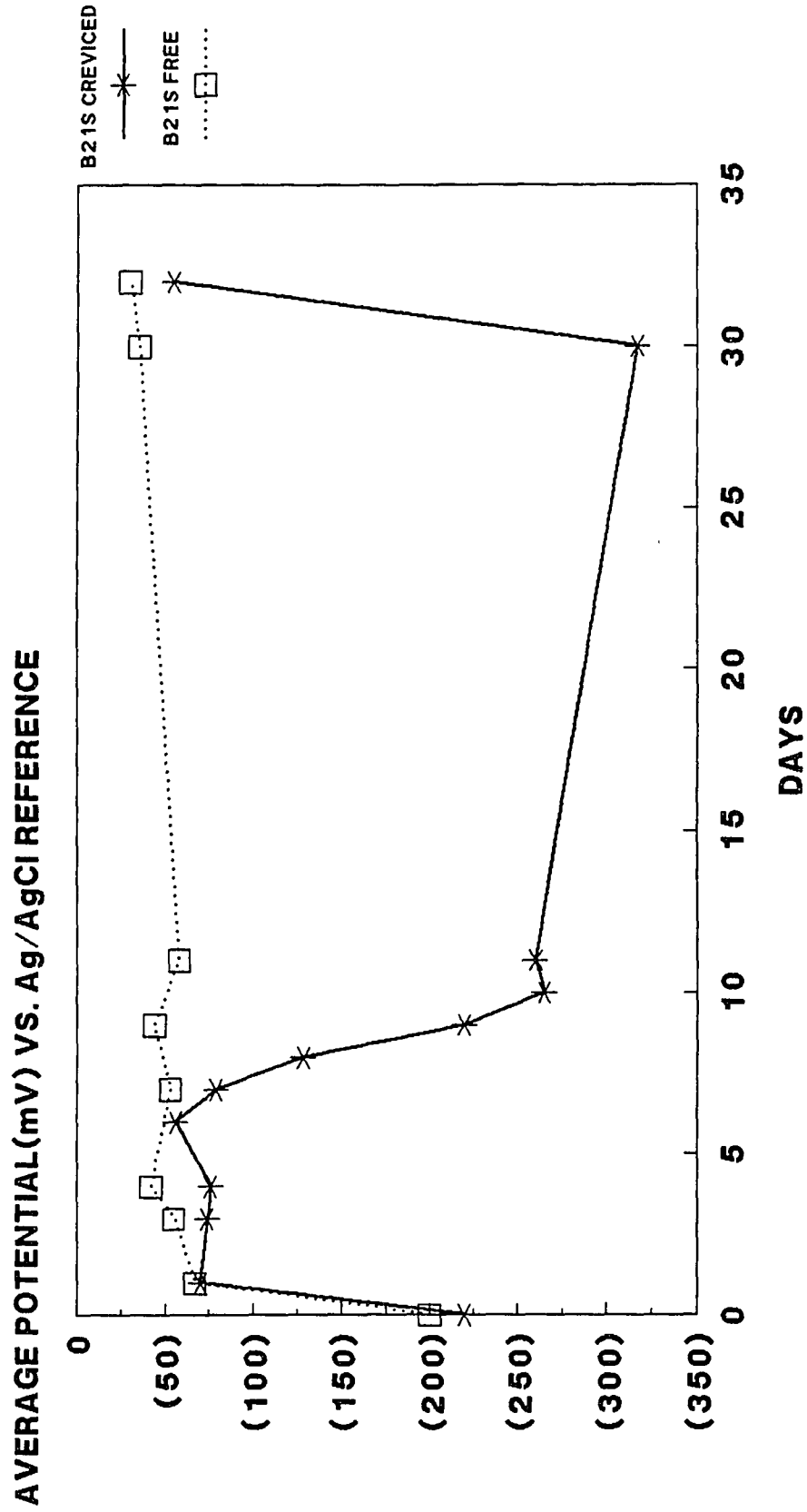


CREVICE CORROSION OBSERVED ON BETA C AT -304 mV
AFTER 4-5 DAYS. WHEN CORROSION INITIATED PARTIAL
PASSIVATIONS WERE DUE TO CORR. PRODUCT OXIDIZERS

5% NaCl, PH 0.5
95 DEGREE C

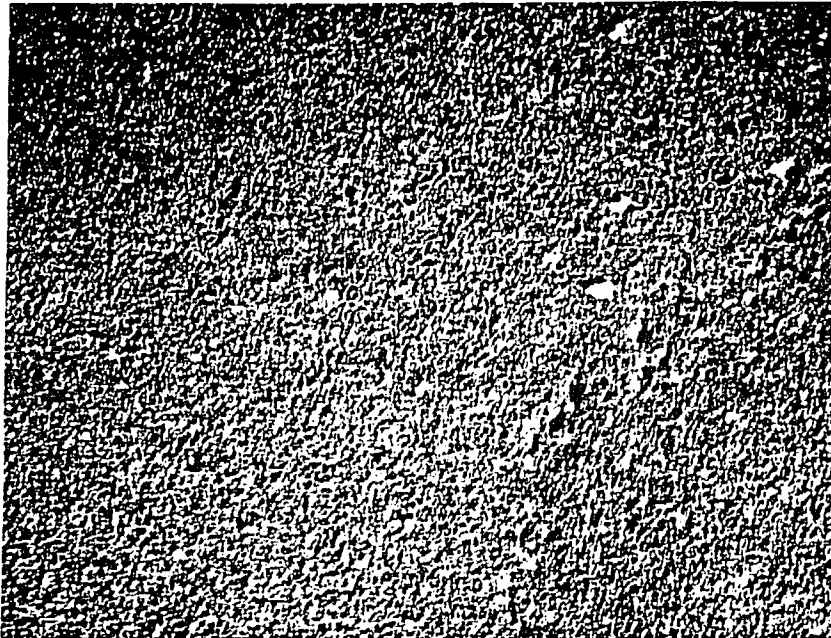
Figure 9c

CREVICE CORROSION POTENTIAL
TITANIUM BETA-21S
PH 0.2

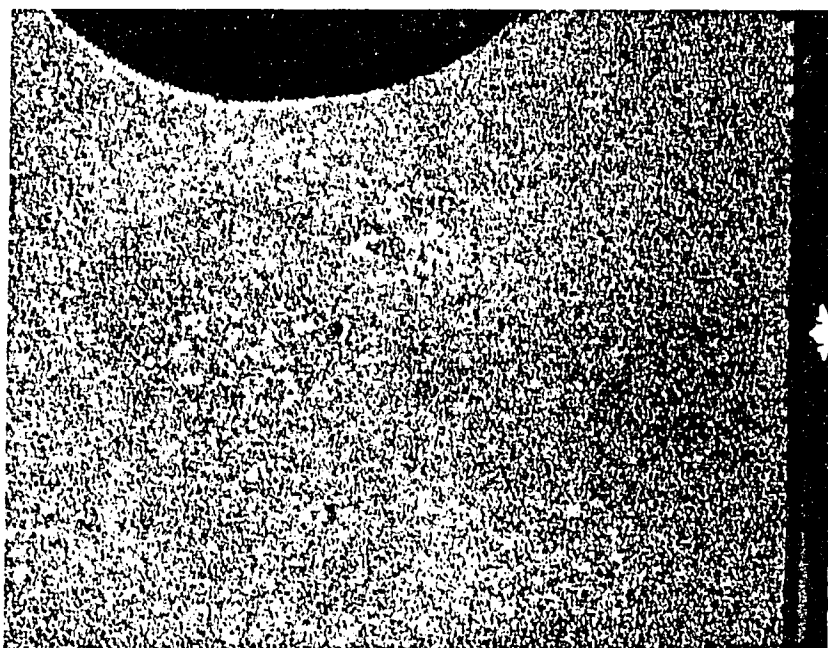


B21S HAD SURFACE CORROSION AT -317mV, 30 DAYS
CREVICE CORROSION WAS NOT OBSERVED.

5% NaCl, PH 0.2
98 DEGREE C

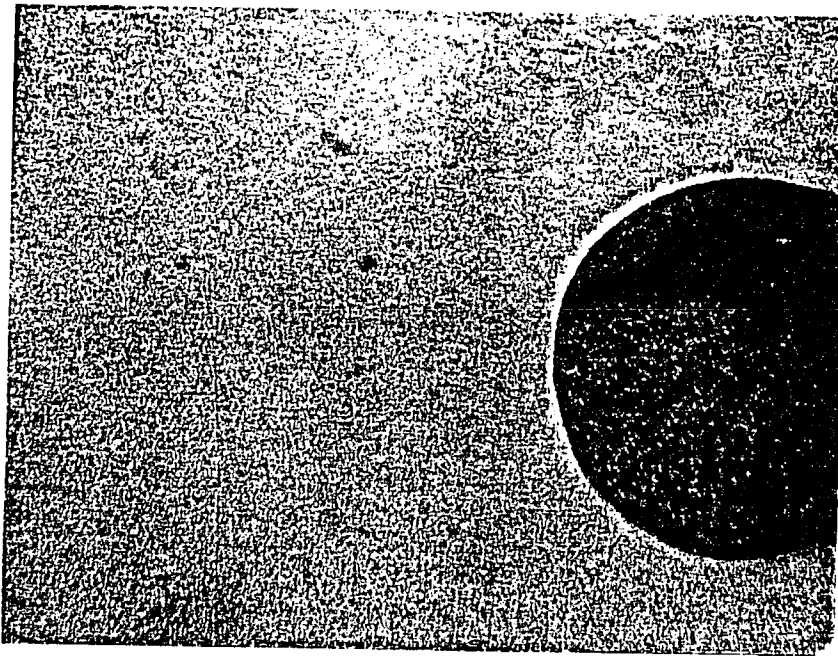


Grade 1
-190 mV
M4395
16X

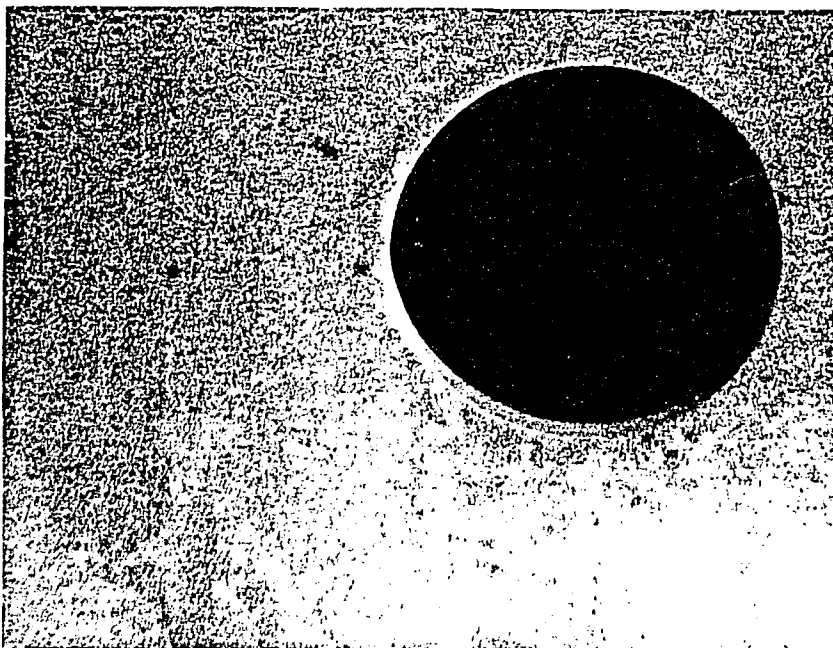


Grade 2
-290 mV
M4393
16X

Figure 10a. Crevice/Uncreviced Ratio 4:1, 5% NaCl, pH 2, 93°C.

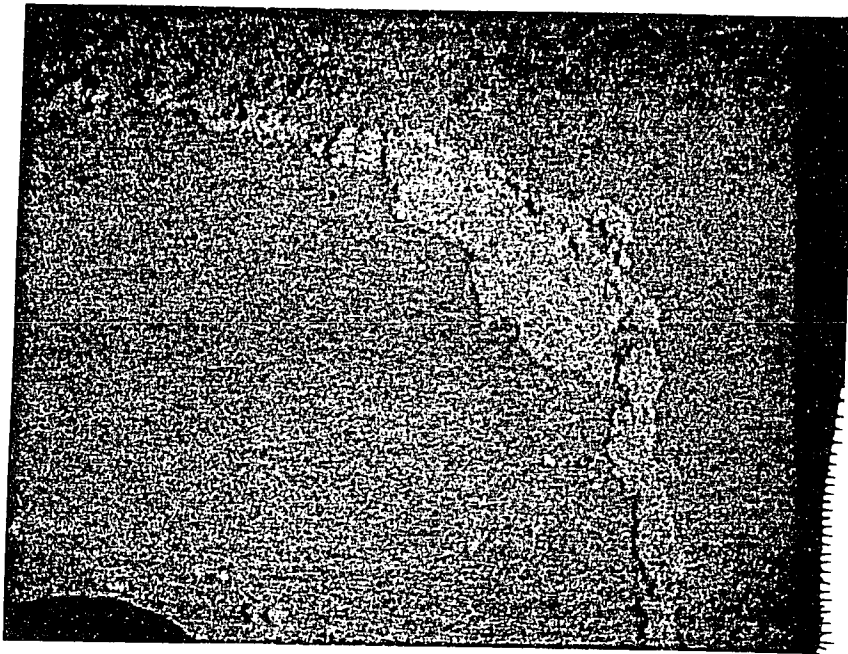


Grade 1
-300 mV
M4398
8X

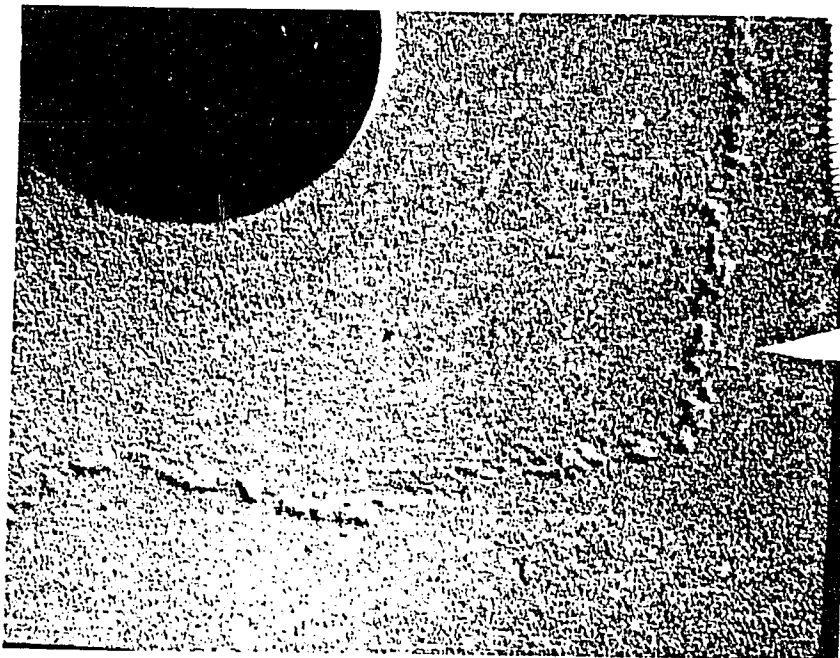


Grade 2
-316 mV
M4399
8X

Figure 10b. Crevice/Uncreviced Ratio 4:1, 5% NaCl, pH 2, 93°C.

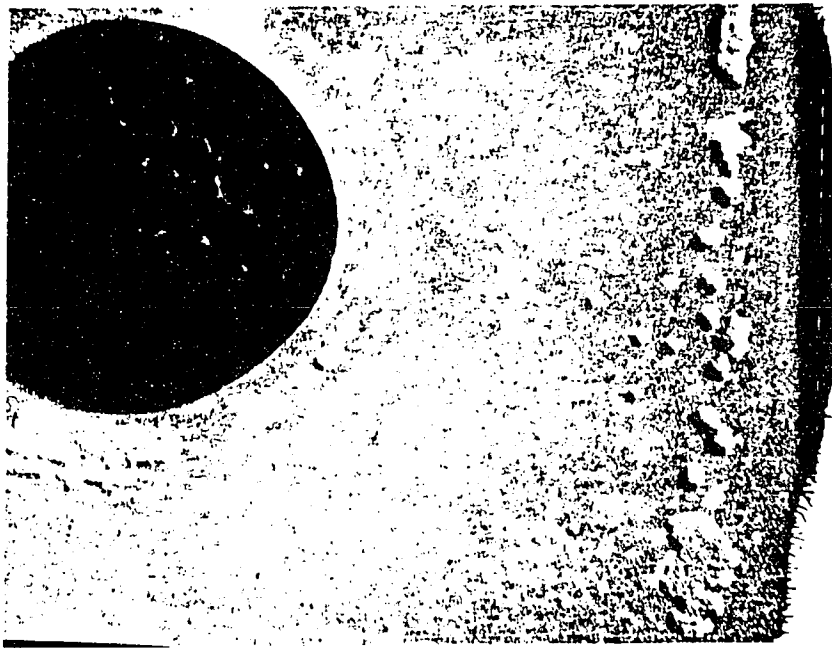


Grade 1
-365 mV
M4400
8X

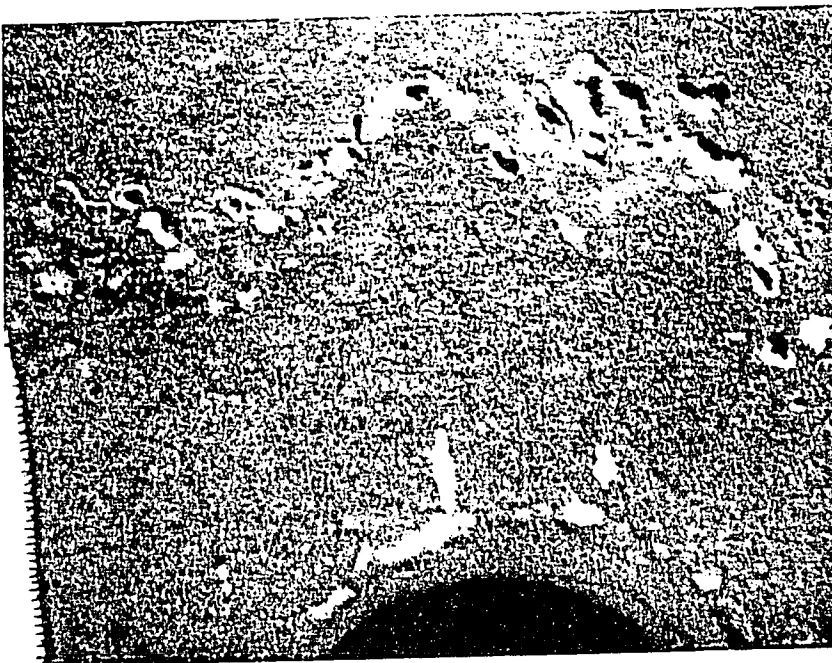


Grade 2
-378 mV
M4346
8X

Figure 10c. Crevice/Uncreviced Ratio 4:1, 5% NaCl, pH 2, 93°C.



Grade 1
-420 mV
M4345
8X

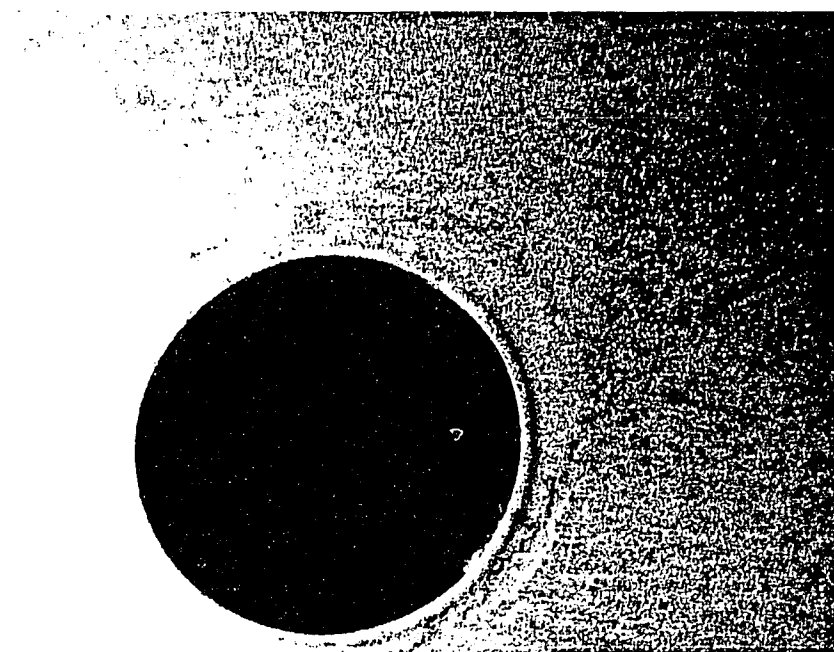


Grade 2
-400 mV
M4347
8X

Figure 10d. Crevice/Uncreviced Ratio 4:1, 5% NaCl, pH 2, 93°C.

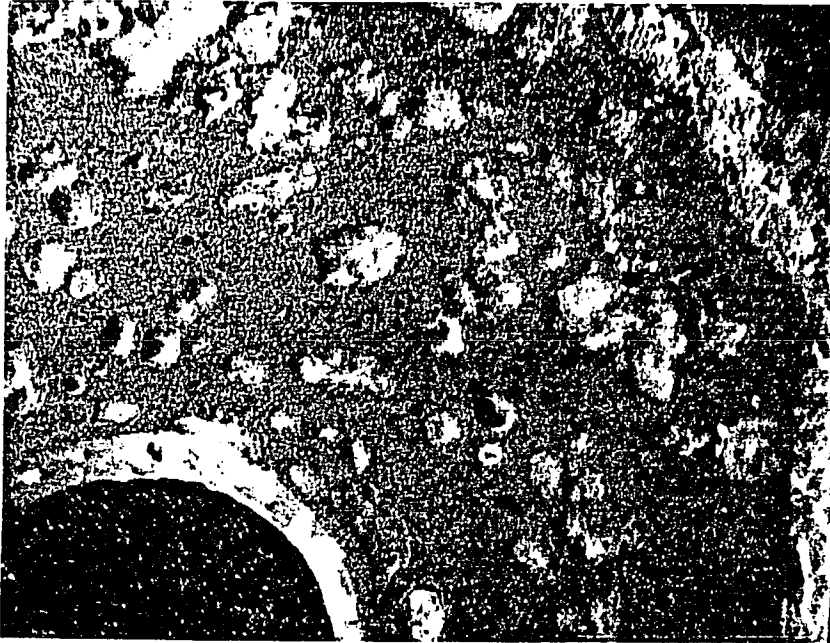


Grade 1
-320 mV
M4402
8X

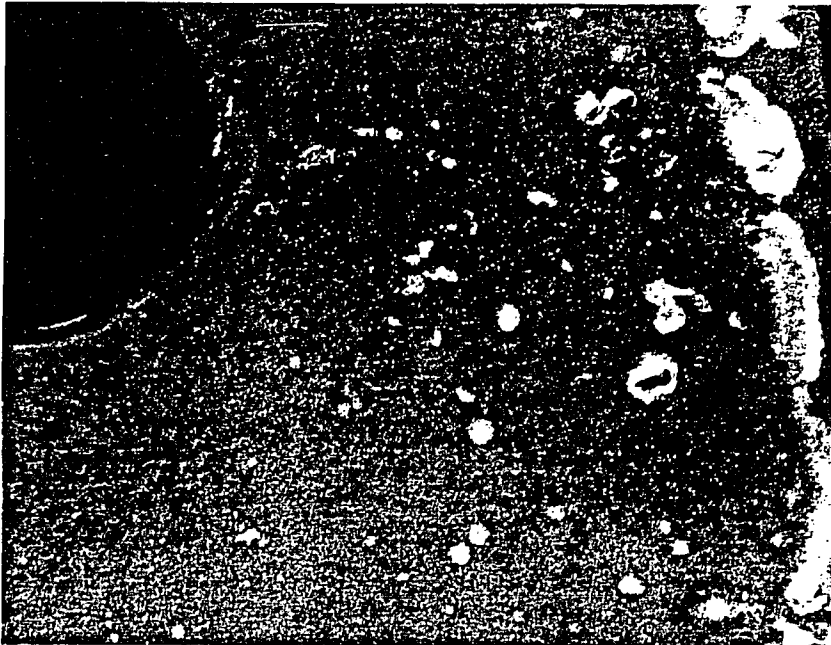


Grade 2
-345 mV
M4401
8X

Figure 11a. Crevice/Uncreviced Ratio 4:1, 5% NaCl, pH 2, 93°C.

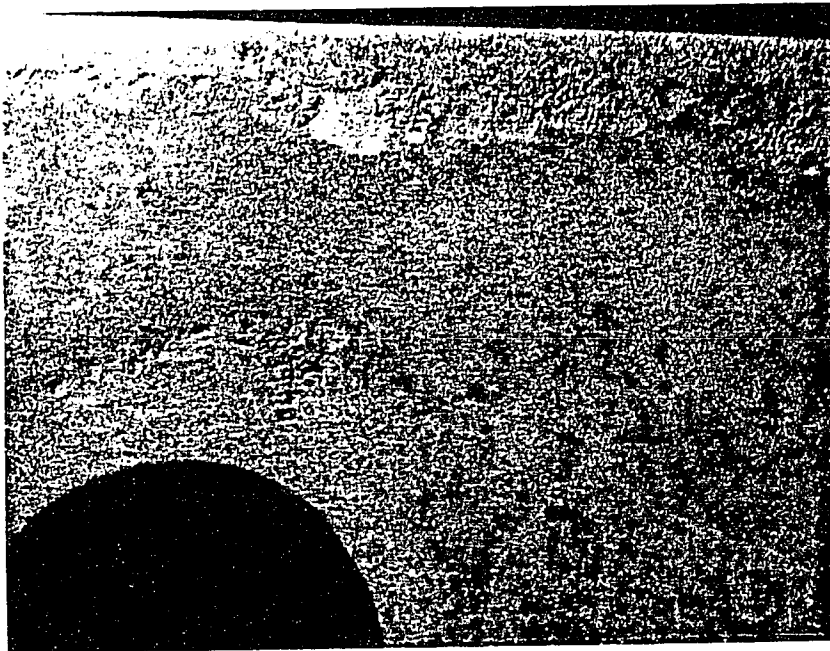


Grade 1
-440 mV
M4404
8X

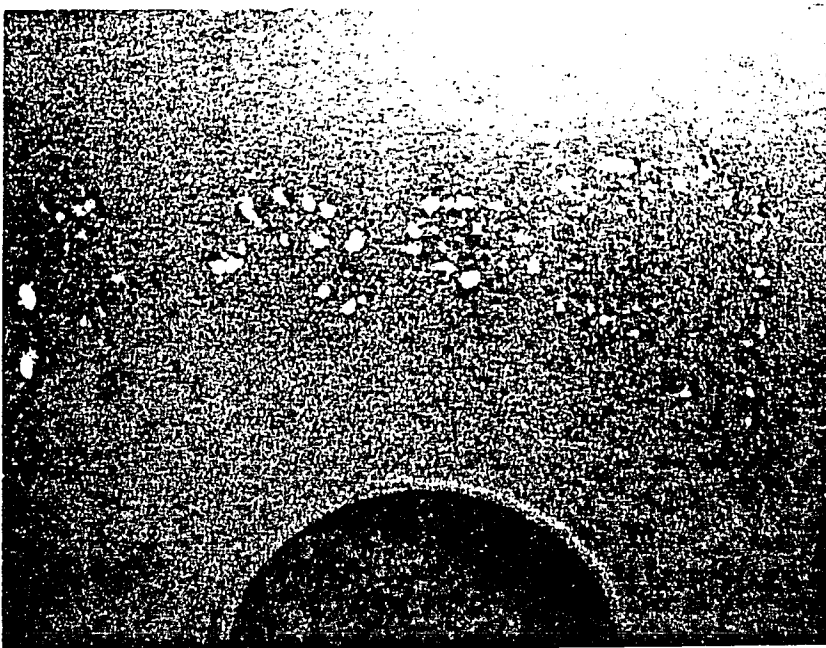


Grade 2
-440 mV
M4403
8X

Figure 11b. Crevice/Uncreviced Ratio 4:1, 5% NaCl, pH 2, 93°C.

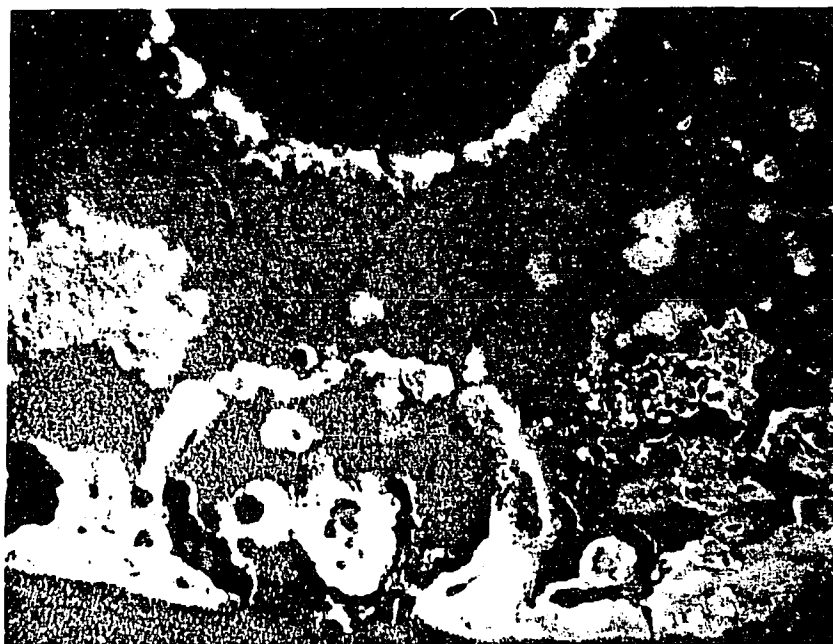


Grade 1
-390 mV
M4405
8X

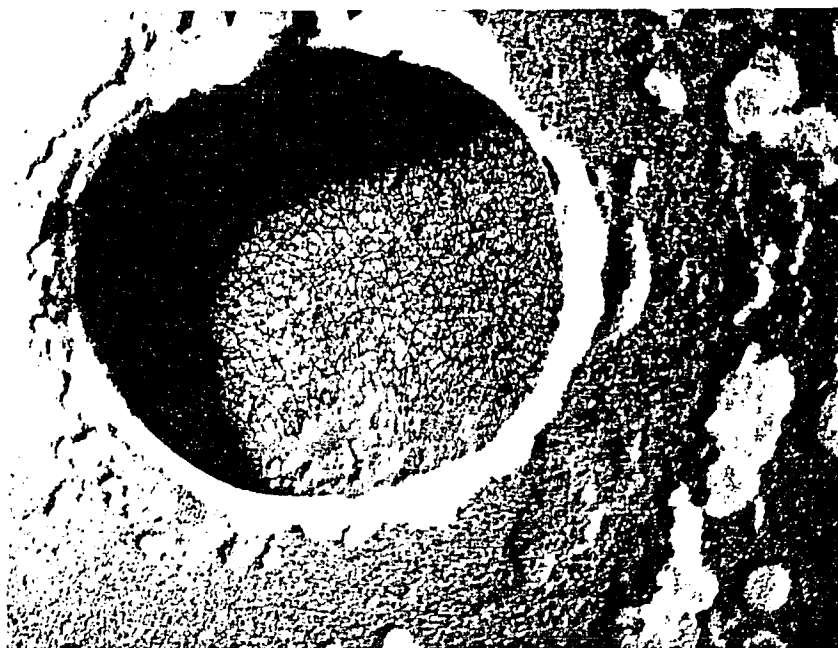


Grade 2
-365 mV
M4406
8X

Figure 12a. Crevice/Uncreviced Ratio 4:1 With Air Sparging,
5% NaCl, pH 2, 93°C.

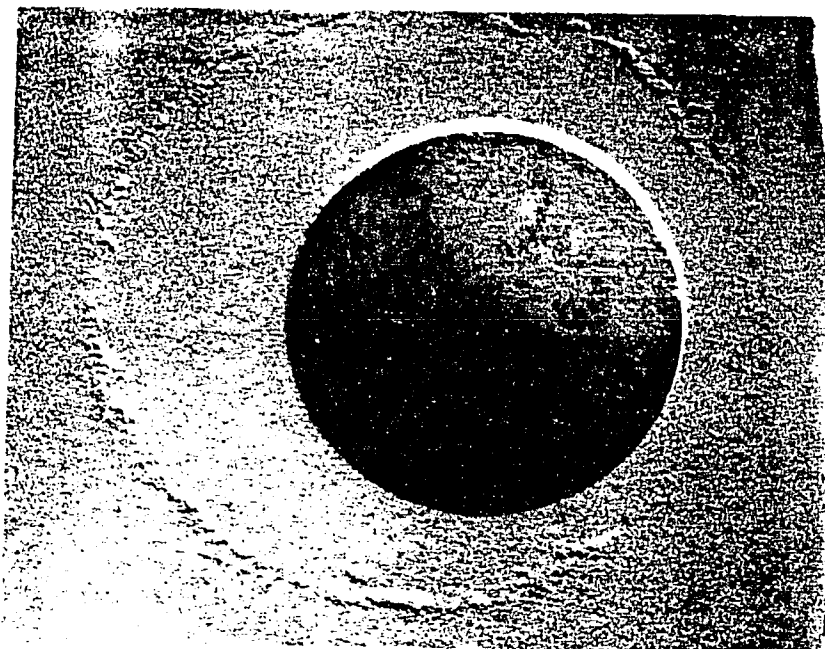


Grade 1
-480 mV
M4407
8X

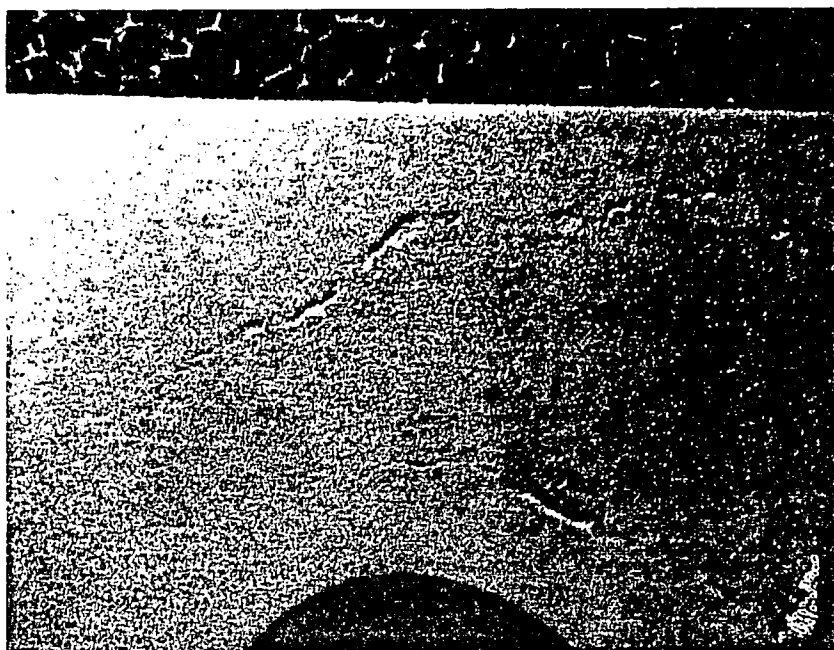


Grade 2
-485 mV
M4408
8X

Figure 12b. Crevice/Uncreviced Ratio 4:1 With Air Sparging,
5% NaCl, pH 2, 93°C.

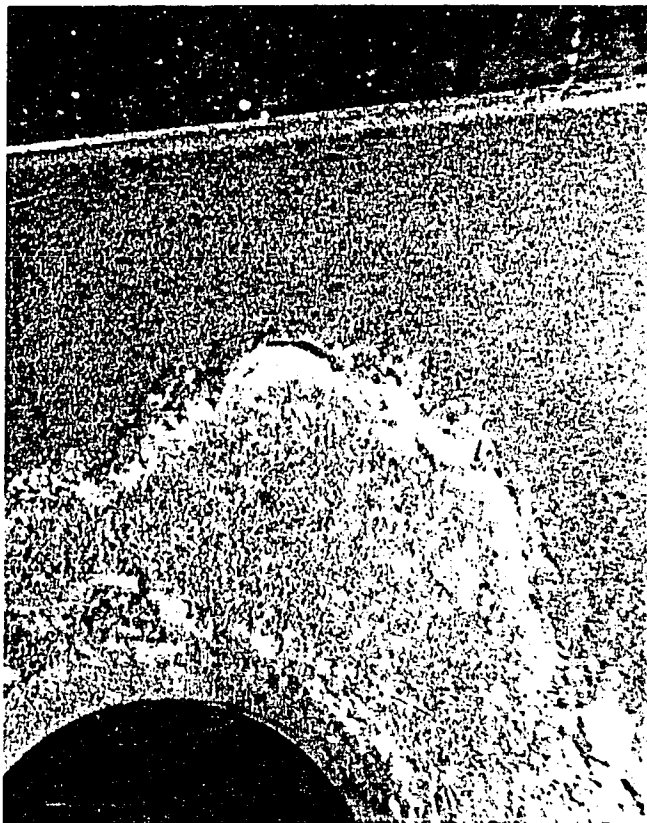


Grade 1
+517 mV
M4411
8X



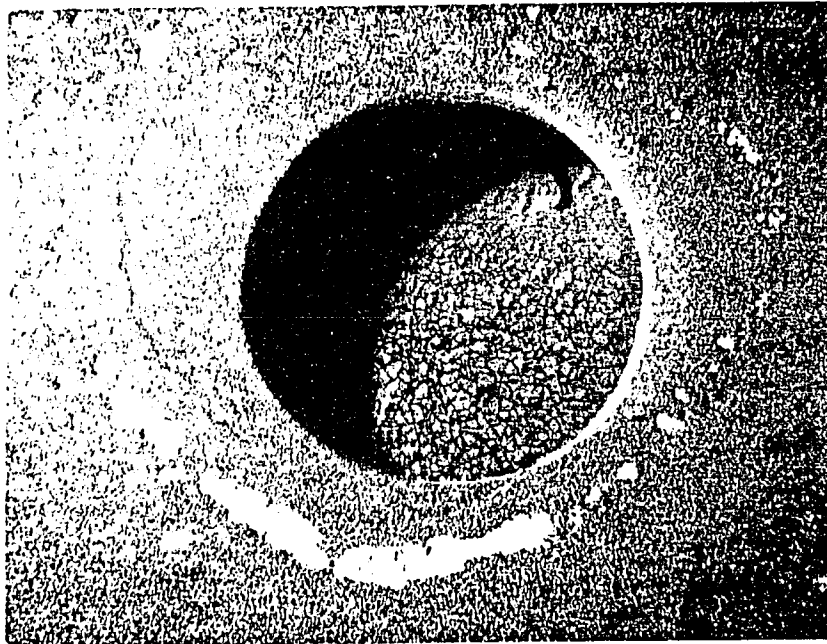
Grade 2
+503 mV
M4412
8X

Figure 13a. 1000 ppm Fe^{+3} in Bulk Solution, Crevice Solution Without Fe^{+3} . 5% NaCl, pH 2, 93°C.

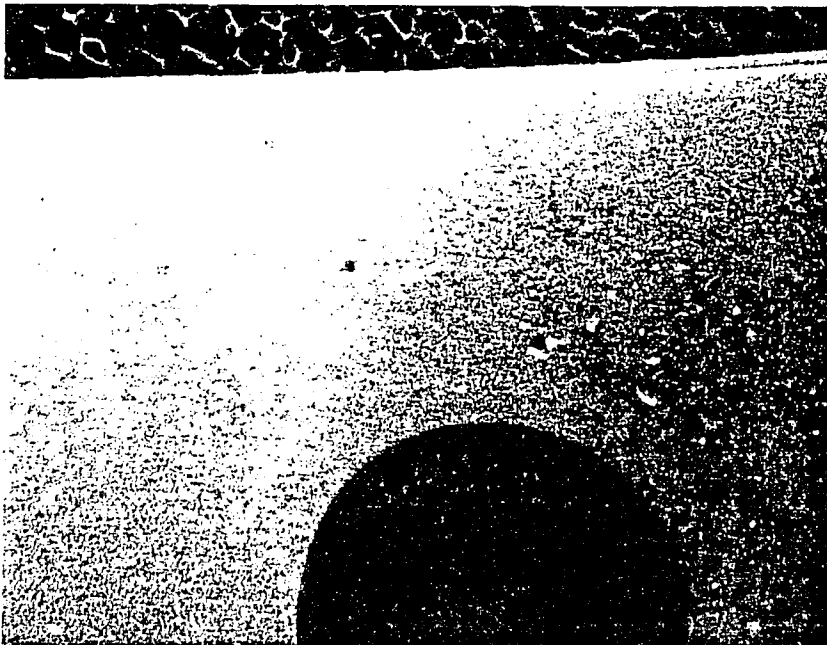


Grade 1
350 mV
M4413
8X

Figure 13b. 1000 ppm Fe^{+3} in Bulk Solution, Crevice Solution Without Fe^{+3} . 5% NaCl, pH 2, 93°C.



Grade 1
-365 mV
M4416
8X



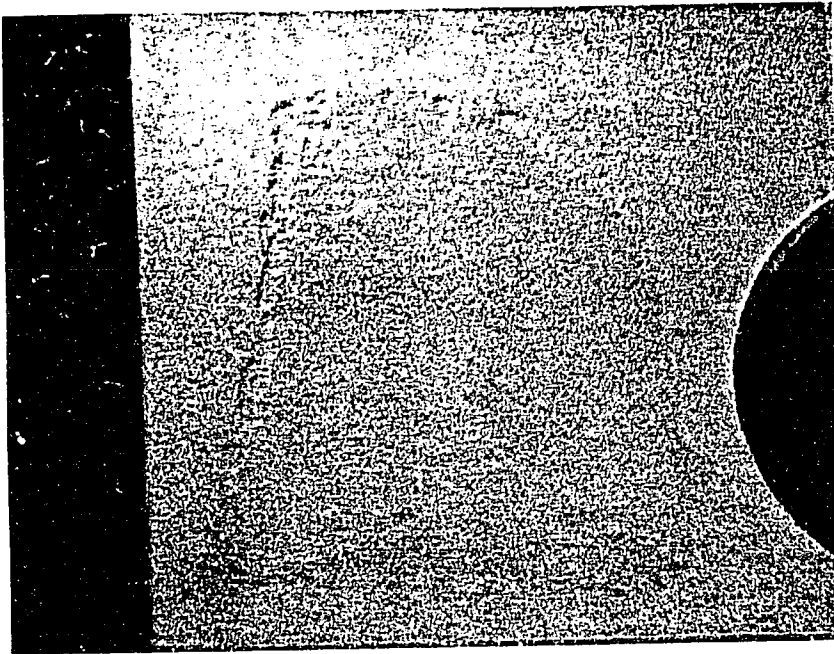
Grade 2
-346 mV
M4414
8X

Figure 14a. Crevice/Uncreviced Ratio 4:1, 5% NaCl, pH 5, 93°C.

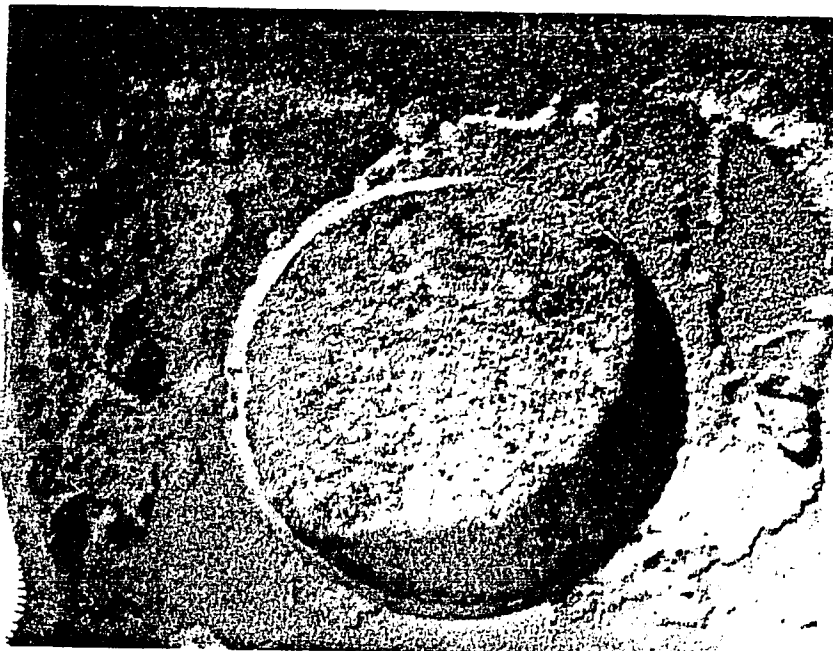


Grade 1
-420 mV
M4415
8X

Figure 14b. Crevice/Uncreviced Ratio 4:1, 5% NaCl, pH 5, 93°C.

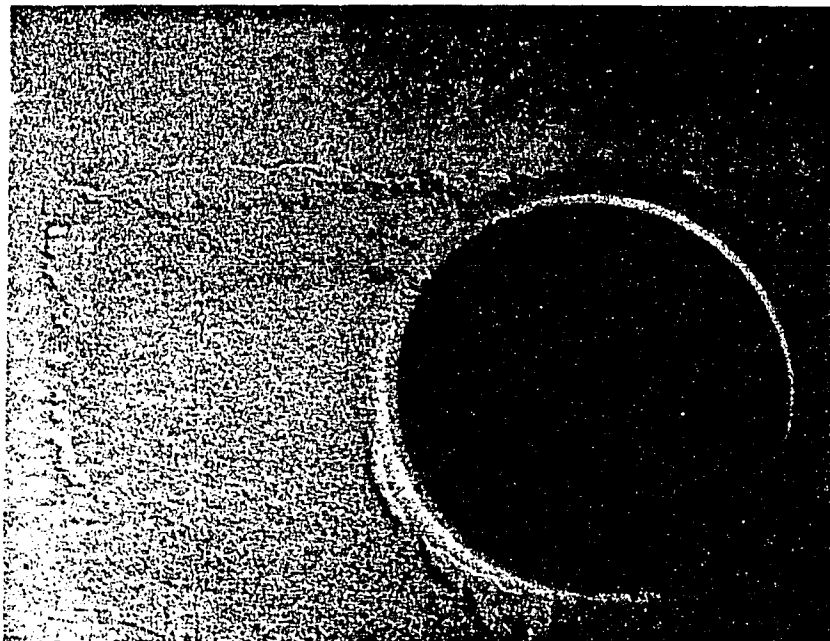


Grade 1
-340 mV
M4279
8X

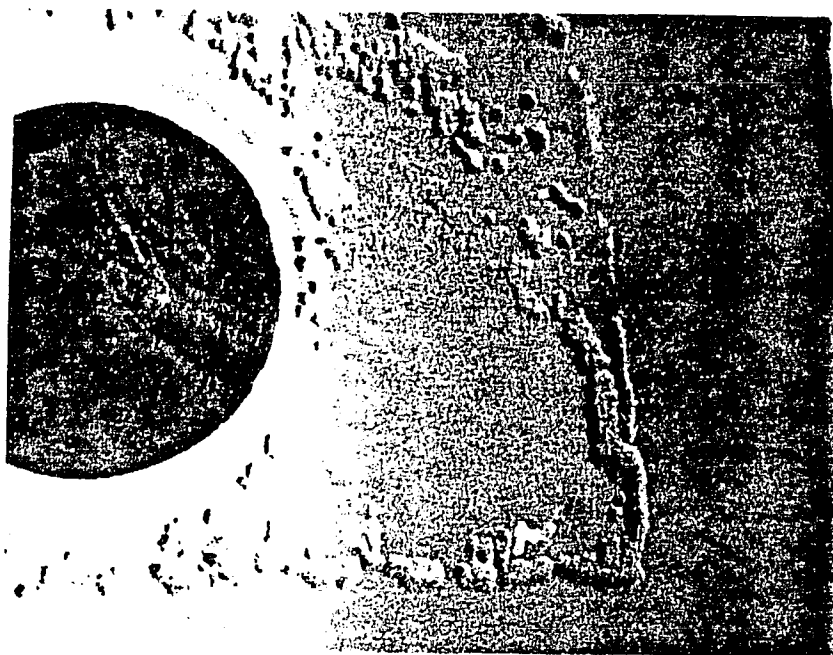


Grade 1
-480 mV
M4296
8X

Figure 15. Crevice/Uncreviced Ratio 1:3, 5% NaCl, pH 2, 93°C.



Grade 1
-300 mV
M4292
8X



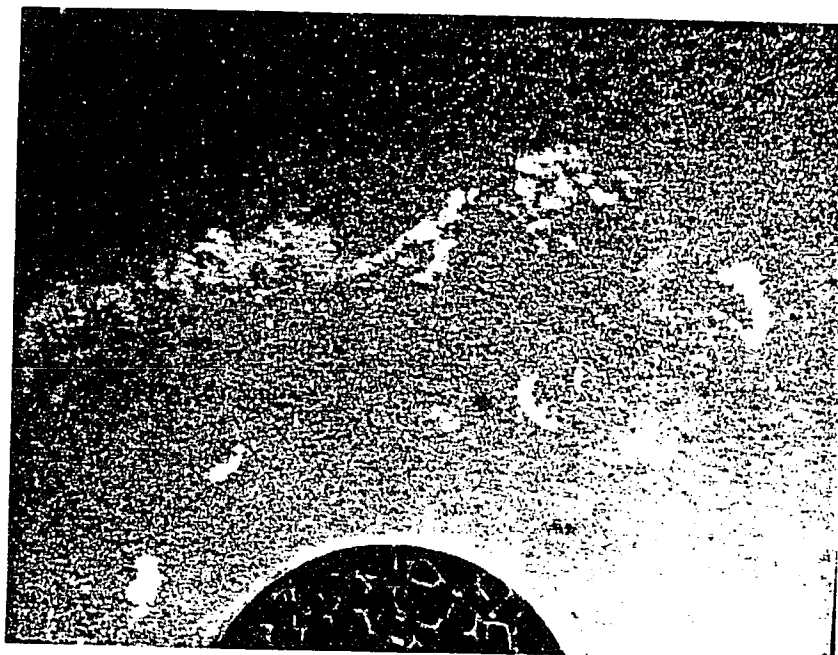
Grade 1
-260 mV
M4417
8X

Figure 16a. Crevice/Uncreviced Ratio 1:90, 5% NaCl, pH 2, 93°C.



Grade 1
-480 mV
M4418
8X

Figure 16b. Crevice/Uncreviced Ratio 1:90, 5% NaCl, pH 2, 93°C.

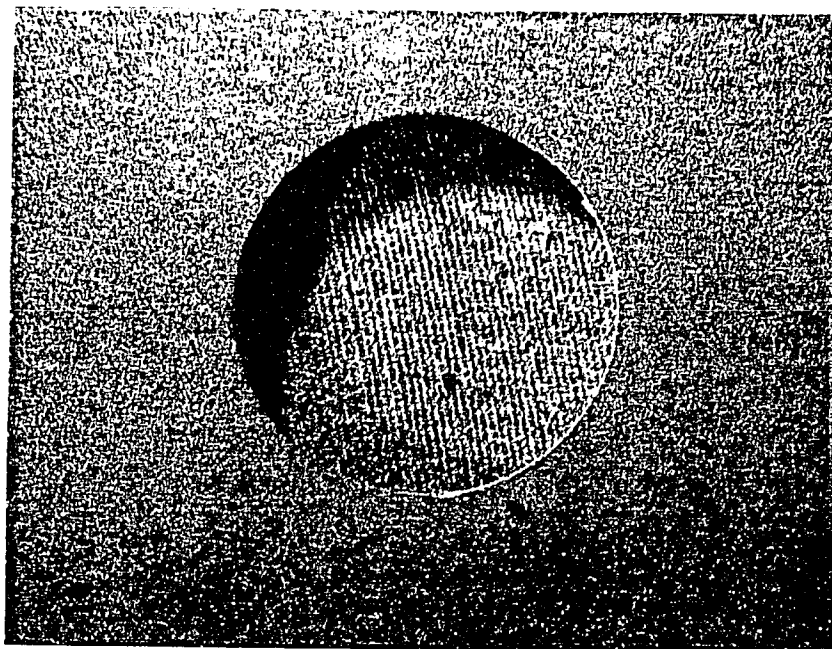


Grade 12
pH 1
-158 mV
M4419
8X

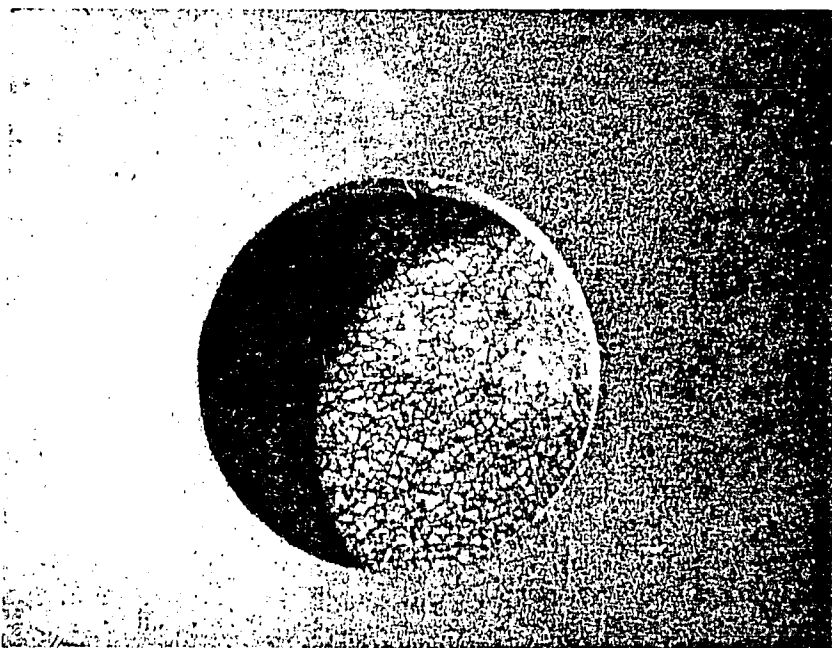


Grade 12
pH 0.5
-160 mV
M4420
8X

Figure 17. Grade 12, 5% NaCl, 95°C, Crevice/Uncreviced Ratio 4:1.

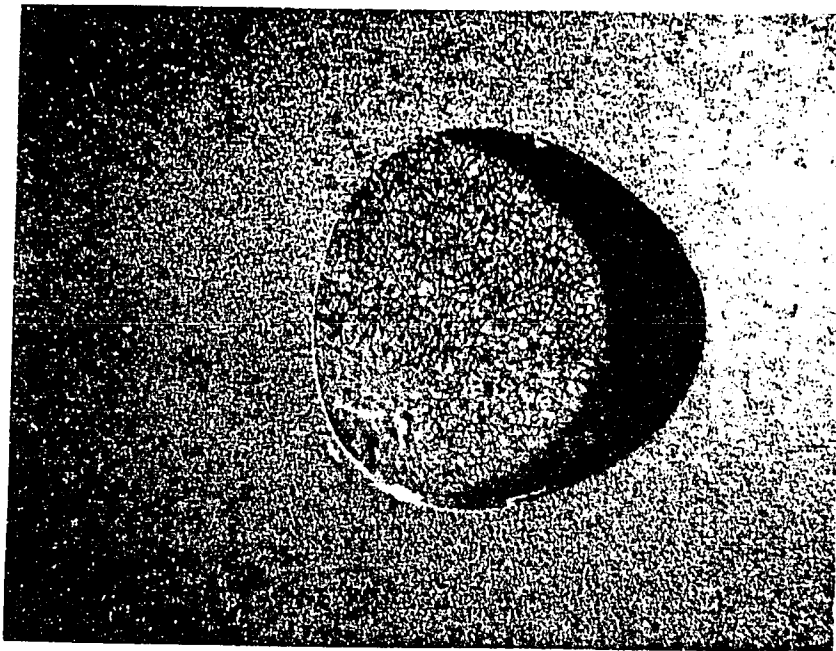


Grade 7
pH 1
+425 mV
M4421
8X

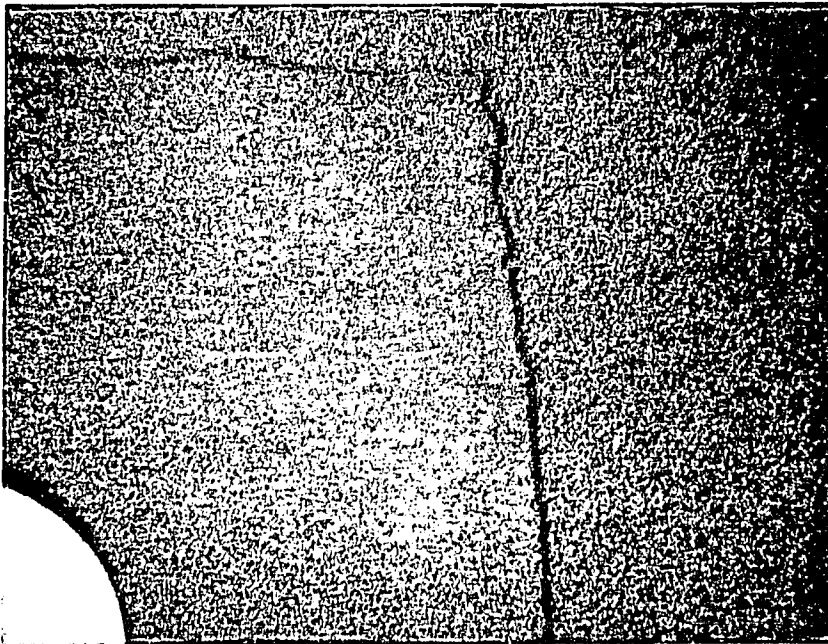


Grade 7
pH 0.5
+210 mV
M4422
8X

Figure 18. Grade 7, 5% NaCl, 95°C, Crevice/Uncreviced Ratio 4:1.

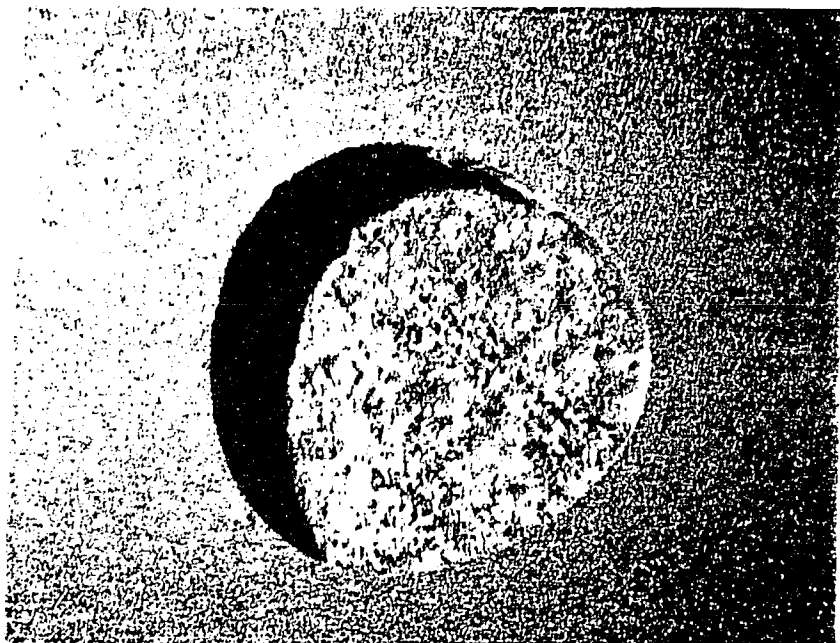


Grade 2
+220 mV With
0.01 Wt.% Pd
pH 1
M4423
8X

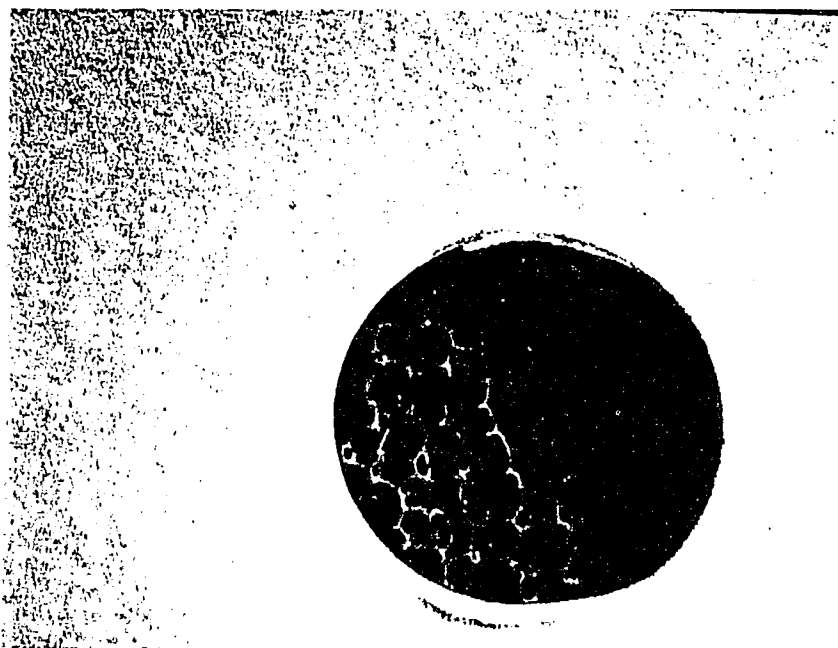


Grade 2
-290 mV With
0.01 Wt.% Pd
pH 0.5
M4363
8X

Figure 19a. Grade 2 0.01, 0.02, 0.05 Pd Wt.%.
Crevice/Uncreviced Ratio 4:1, 5% NaCl, 95°C.

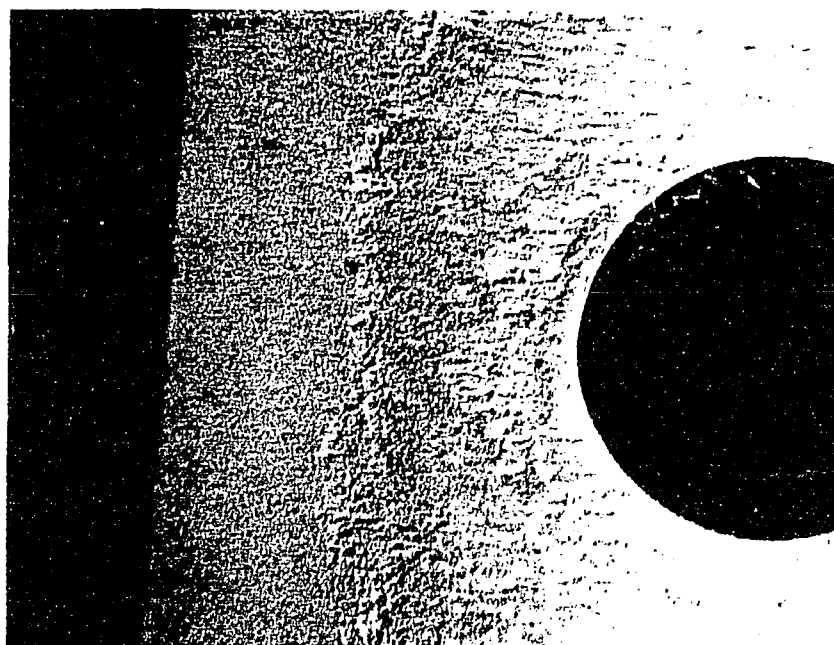


Grade 2
0.02% Wt. Pd
pH 0.5
-295 mV
M4341
8X

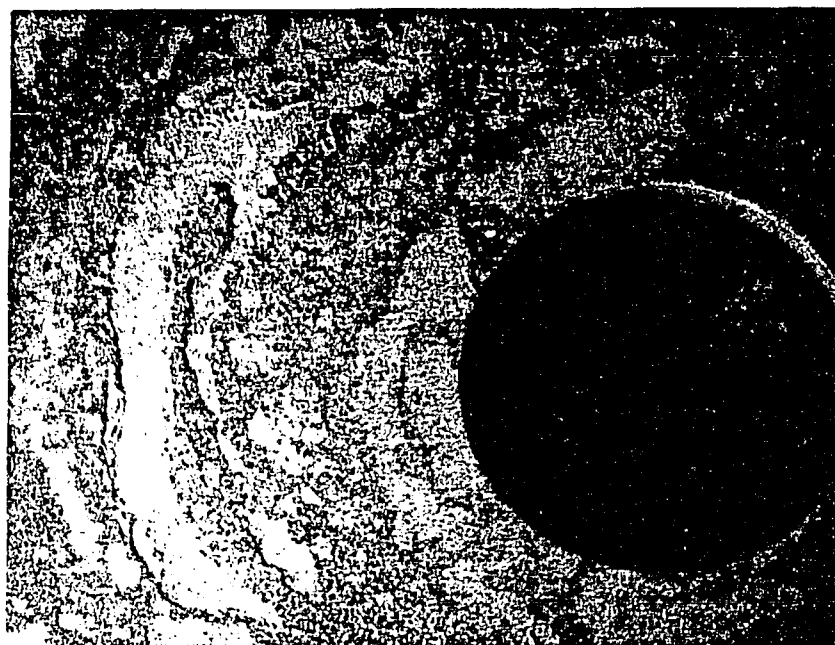


Grade 2
0.05% Wt. Pd
pH 0.5
-290 mV
M4344
8X

Figure 19b. Grade 2, 5% NaCl, 95°C, Crevice/Uncreviced Ratio 4:1.

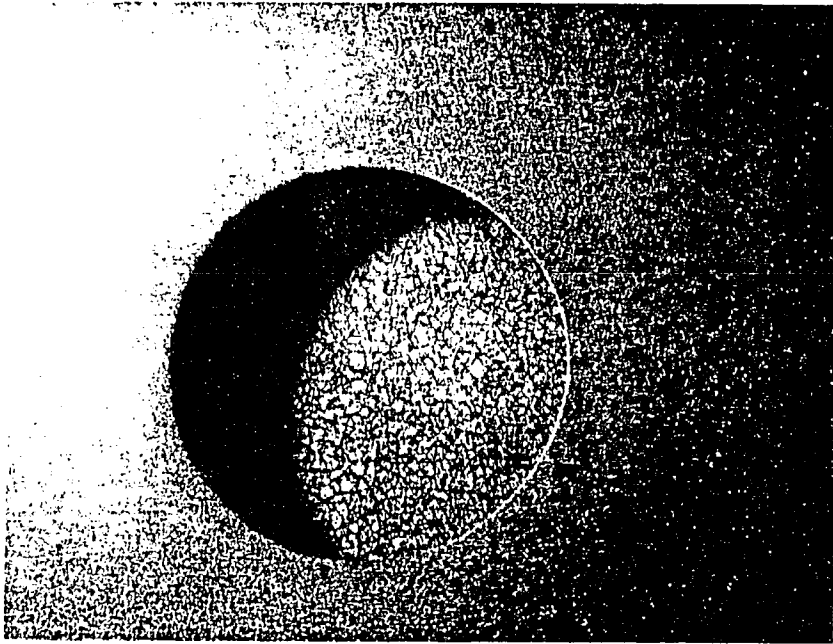


Ti-3Al-8V-6Cr-4Mo-4Zr
(Beta C)
pH 1
-304 mV
M4424
8X

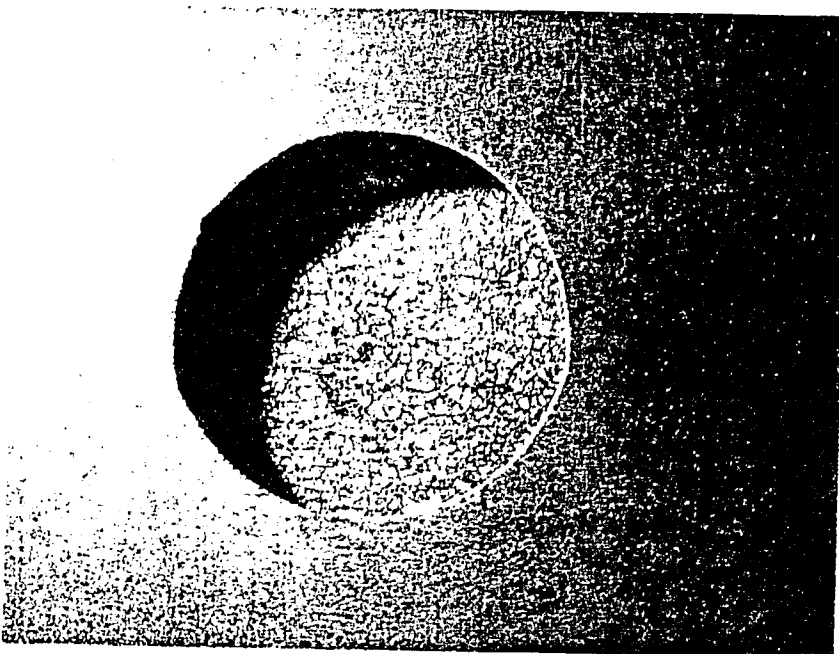


Ti-3Al-8V-6Cr-4Mo-4Zr
(Beta C)
pH 0.5
-368 mV
M4425
8X

Figure 20. Ti-3Al-8V-6Cr-4Mo-4Zr (Beta C) Crevice/Uncreviced Ratio 4:10,
5% NaCl, 95°C.

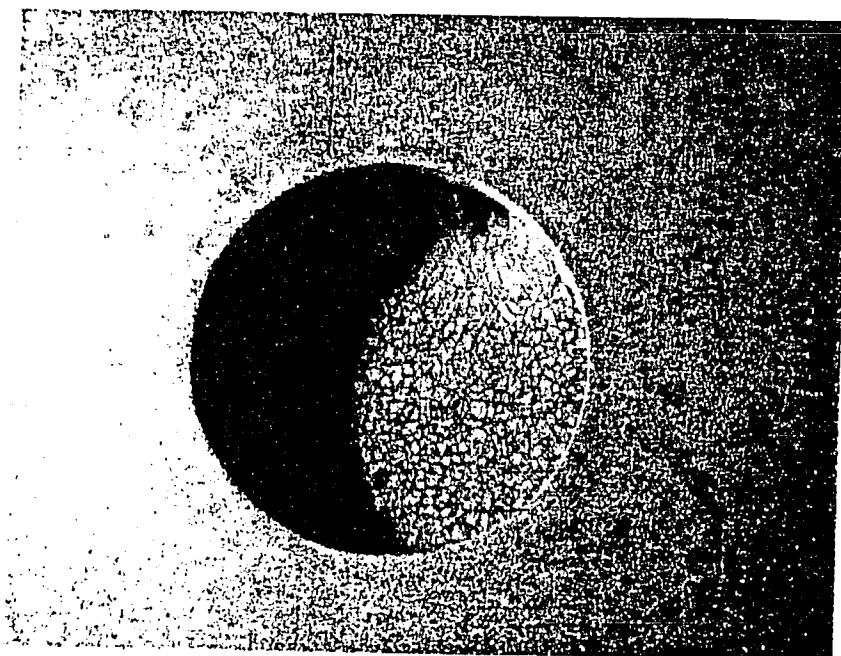


Beta-21S
pH 1
-70 mV
M4426
8X



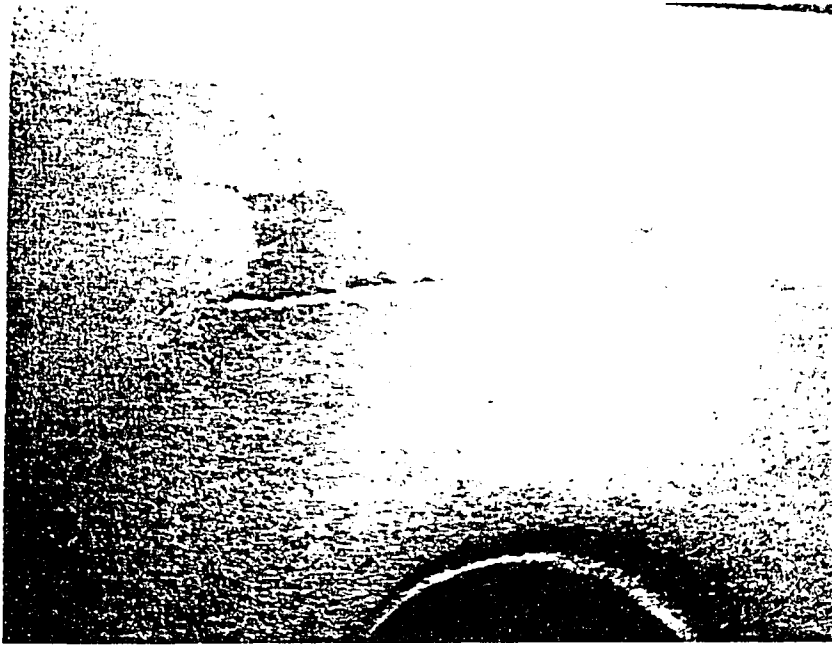
Beta-21S
pH 0.5
+80 mV
M4427
8X

Figure 21a. Beta-21S, Crevice/Uncreviced Ratio 4:1, 5% NaCl, 95°C.



Beta-21S
pH 0.2
-308 mV
M4428
8X

Figure 21b. Beta-21S, Crevice/Uncreviced Ratio 4:1, 5% NaCl, 98°C.

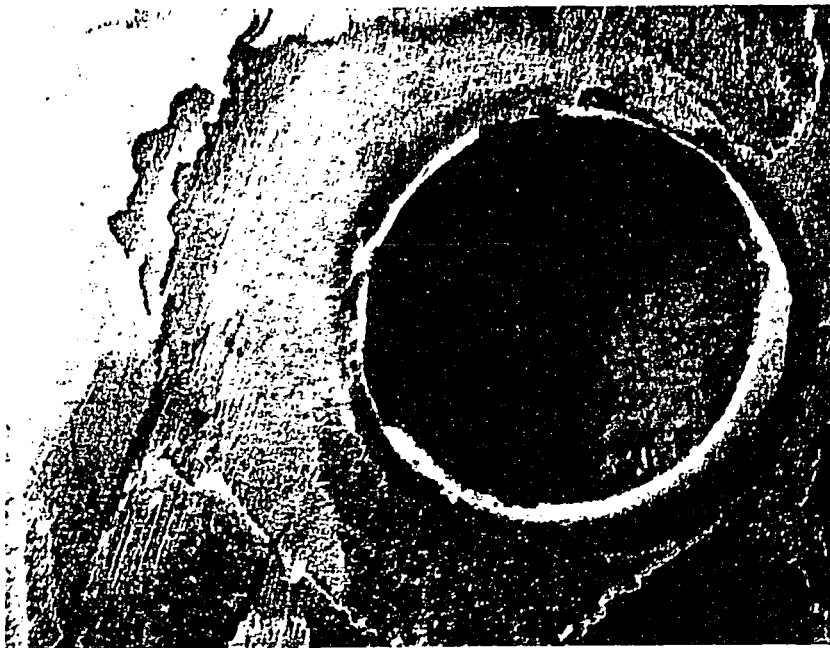


Nickel Alloy 625
pH 2
-267 mV
M4343
8X



Nickel Alloy 625
pH 1
-315 mV
M4342
8X

Figure 22a. Nickel Alloy 625 Crevice/Uncreviced Ratio 4:1, 5% NaCl, 95°C.



Nickel Alloy 276
pH 1
-300 mV
M4397
8X



Nickel Alloy 276
pH 0.5
-310 mV
M4368
8X

Figure 22b. Nickel Alloy 625 Crevice/Uncreviced Ratio 4:1, 5% NaCl, 95°C.